1 of 121

ROY F. WESTON, INC.

QUALITY ASSURANCE PROJECT PLAN
AMENDMENT NO. 2 – AIR SAMPLING ACTIVITIES
EVERGREEN MANOR
ROSCOE, ILLINOIS

WORK ASSIGNMENT NO. 139-RICO-05MZ

REVISION 1 – 22 MAY 2002

WESTON
MANAGERS DESIGNERS CONSULTANTS

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Prepared for:

U.S. Environmental Protection Agency
Region V
77 West Jackson Boulevard
Chicago, Illinois 60604

This document was prepared in accordance with U.S. EPA Contract No. 68-W7-0026,WESTON Region V Response Action Contract

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22 May 2002

Ms. Karen Cibulskis (SR-6J)
Remedial Project Manager
United States Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604

U.S. EPA Contract No.: 68-W7-0026
Work Assignment No.: 139-RICO-05MZ
Document Control No.: RFW139-2E-ALCI

Subject: QAPP and FSP Amendment No. 2, Revision 1

Dear Ms. Cibulskis:

Roy F. Weston, Inc. (WESTON®) is pleased to submit the responses to U.S. EPA Comments (dated 24 April 2002) on the QAPP Amendment No. 2 (Revision 0) and QAPP Amendment No. 2 (Revision 1). This QAPP Amendment relates to the planned air sampling activities at the Evergreen Manor Site.

Should you have any questions or require additional information, please feel free to contact me at (847) 918-4005.

Very truly yours,

ROY F. WESTON, INC.

Deepak L. Bhojwani
Site Manager

DLB:ld
Enclosures

cc: James M. Burton, P.E. (WESTON)

Response to U.S. EPA Comments
Quality Assurance Project Plan
Amendment No. 2, Revision 0
Evergreen Manor Site
Roscoe, Illinois

GENERAL COMMENTS

U.S. EPA Comment 1: *Please make sure that all of the sections in the QAPP are included and labeled.*

WESTON Response: The air sampling activities task is being handled as a QAPP Amendment. This is Amendment 2 to the original Evergreen Manor QAPP/FSP. In accordance with U.S. EPA requirements, only those sections and portions of the QAPP which deviated from the original QAPP were submitted as part of the Amendment. The original QAPP/FSP numbering scheme was utilized whenever possible.

U.S. EPA Comment 2: *The soil gas samples need to be taken at a depth below the foundation of the house - 3 feet is too shallow. Please revise the QAPP to specify how this will be determined.*

WESTON Response: WESTON will utilize the U.S. EPA GeoProbe to increase the depth at which the soil gas samples will be collected. WESTON will collect the soil gas samples from depths of 6 to 8 feet below ground surface, or below the foundation, whichever is shallower. If the underlying geology does not allow for deeper penetration, samples will be collected at the deepest point possible, not less than 3 feet below ground surface.

U.S. EPA Comment 3: *U.S. EPA's air expert (Mr. Dave Mikhunas) has suggested that we collect soil gas samples at a minimum of 4 locations around each house. Dave has said that he has seen 3 orders of magnitude variation around houses that he has sampled. Please respond and discuss whether Weston believes this approach is necessary or not.*

WESTON Response: The original number of samples proposed by WESTON was determined based on the original request by the U.S. EPA. WESTON is in agreement with Mr. Mikhunas's assessment. WESTON will collect 4 soil gas samples around each of the four houses.

U.S. EPA Comment 4: *Purge 3 volumes from the soil gas sampling probes. This will clear the lines and not deplete the soil gas. Please revise the QAPP.*

WESTON Response: WESTON will purge 3 volumes from the soil gas sampling probes when depths of 3 to 8 feet below ground surface are achieved.

U.S. EPA Comment 5: *U.S. EPA's air expert has suggested that we place Summa canisters in the basement and on the first floor of each house to help determine if the source of any detected chemicals is from lifestyle or subsurface contamination. Please respond and discuss whether Weston believes this approach is necessary or not.*

WESTON Response: The original number of samples proposed by WESTON was determined based on the original request by the U.S. EPA. WESTON is in agreement with Mr. Mikhunas's assessment. WESTON will collect a sample in the basement and on the first floor of each house. WESTON feels that by collecting the first floor sample, a gradient, if any, can be determined for contaminants found in the samples. If a target compound is detected in a basement sample, the first floor sample will help to evaluate whether it could have originated from an alternate activity in the home.

U.S. EPA Comment 6: *Do not conduct the sampling after a major rain event - the water fills the interstitial spaces and lowers the results - have meteorological data collected before and during the sampling. Please revise the QAPP to indicate this.*

WESTON Response: WESTON will not collect air samples after a major rain event. WESTON will coordinate with the residences and the laboratory in the event that a postponement of sampling activities is warranted.

U.S. EPA Comment 7: *The detection limits for your target compounds are below the cleaning levels stated in the TO Methods - please have all canisters certified clean to the detection limit levels. Please revise.*

WESTON Response: The laboratory has stated that the canisters will be cleaned to the detection levels specified for this project in Table 4-4.

U.S. EPA Comment 8: *U.S. EPA's air expert is not certain if the canisters have been shown to be stable in the part per trillion level for these compounds. Please verify and respond.*

WESTON Response: The laboratory method detection limit studies show that canisters would be stable in the part per trillion level for the compounds of interest. Tables associated with these studies will be referenced in the QAPP Amendment, Revision 1 and included for review.

U.S. EPA Comment 9: *U.S. EPA's air expert suggests that we might want to first screen the homes with the TAGA. Please consider and respond.*

WESTON Response: WESTON feels that the comprehensive survey questionnaire that will be used prior to sampling together with specific resident instructions, and the specific target compounds list should provide enough screening information for evaluation of the sampling data.

SPECIFIC COMMENTS

U.S. EPA Comment 1: Section 2.3.2, typo, Page 1 of 13: This should be Section 2.4.2 Project Objectives. See QAPP Page 9 of 17.

WESTON Response: The error will be corrected in the QAPP, Revision 2.

U.S. EPA Comment 2: Section 3.1, editorial: For this project the WESTON Site Manager is Deepak L. Bhojwani.

WESTON Response: This update was previously made in QAPP Amendment No. 1. This QAPP Amendment relates to other environmental sampling activities.

U.S. EPA Comment 3: Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, Page 5: Section 6.3 appears to be missing, or Section 6.0 is misnumbered.

WESTON Response: The laboratory has stated that the information in that section is proprietary information and is therefore not submitted.

U.S. EPA Comment 4: Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, Section 9.2: Specify the concentrations of the 5 calibration levels.

WESTON Response: Initial calibration data has been supplied by the laboratory and will be included with Revision 1 QAPP Amendment.

U.S. EPA Comment 5: Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, Appendix A Table A-1: The project required Compound 2-butanone is not included in this table. See QAPP Table 4-4.

WESTON Response: This information is included in Table 8-3 of the laboratory NELAP Methods manual. It will be included in Revision 1 QAPP Amendment.

U.S. EPA Comment 6: Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, Appendix A Table A-1: Provide Quantitation Ions for 2-butanone, surrogates, and internal standards.

WESTON Response: This information is included in Table 8-3, 8-4, and 8-5 of the laboratory NELAP Methods manual. These tables will be included in QAPP Amendment, Revision 1.

U.S. EPA Comment 7: *Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, Appendix B: This page is blank. Are the TO-14 list of analytes supposed to be included. See Section 8.1.*

WESTON Response: This information is included in Table 8-2 and 8-3 of the laboratory NELAP Methods manual. These tables will be included in QAPP Amendment, Revision 1.

U.S. EPA Comment 8: *Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, AIR TOXICS LTD. (ATL) FAX , Page 2: The project required compound chloroform is missing from this list. See QAPP Table 4-4.*

WESTON Response: A new table which includes chloroform has been prepared and is being submitted with the QAPP Amendment, Revision 1.

U.S. EPA Comment 9: *Analysis of Volatile Organic Compounds in Summa Polished Canisters by GC/MS Selective Monitoring, METHOD DETECTION LIMIT SUMMARY REPORT: The method detection limits (MDLs) for the project required compounds acetone, methylene chloride, 2-butanone, freon 113, and chloroform were not given. Can ATL achieve the project required MDLs as listed in Table A-1? See QAPP Table 4-4.*

WESTON Response: The laboratory method detection limit studies include all of the project required compounds. These tables will be submitted in the QAPP Amendment, Revision 1.

QUALITY ASSURANCE PROJECT PLAN
AMENDMENT NO.2, REVISION 1
EVERGREEN MANOR
ROSCOE, ILLINOIS

22 May 2002

Prepared By: _____ Date: 5/22/02
Tonya Balla, WESTON
Project Engineer

Approved By: _____ Date: 5/22/02
Deepak L. Bhojwani
Site Manager

Approved By: _____ Date: 5/22/02
James M. Burton, P.E.
for Program Manager

Approved By: _____ Date: 5-22-02
Karen Cibulskis
Remedial Project Manager

Approved By: _____ Date: _____
U.S. EPA Quality Assurance Reviewer

QUALITY ASSURANCE PROJECT PLAN
AMENDMENT NO.2, REVISION 0
EVERGREEN MANOR
ROSCOE, ILLINOIS

10 April 2002

Prepared By: Tonya Balla Date: 4-10-02
Tonya Balla, WESTON
Project Engineer

Approved By: Deepak L. Bhojwani Date: 4/10/02
Deepak L. Bhojwani
Site Manager

Approved By: James M. Burton Date: 4/10/02
James M. Burton, P.E.
Program Manager

Approved By: _____ Date: _____
Karen Cibulskis
Remedial Project Manager

Approved By: Richard L. Smith Date: 4/19/02
U.S. EPA Quality Assurance Reviewer
* Conditional Approval

RFW139-2E-AKXD

MEMORANDUM **SMF-4J**

DATE: April 19, 2002

SUBJECT: Conditional Approval of **Amendment** No. 2 to the Quality Assurance Project Plan (QAPP) for the Fund-Lead **Remedial** Investigation/Feasibility Study (RI/FS) Activities for **Evergreen Manor** in Roscoe, Illinois

FROM: Richard L Byvik
Field Services Section (FSS)

TO: Karen Cibulskis
Remedial Project Manager (RPM)

I recommend conditional approval **of Amendment** No. 2 to the QAPP for the Fund-Lead RI/FS activities at **Evergreen Manor, Roscoe, Illinois**. The subject QAPP was received by FSS on April 11, 2002, Log-in #2876. The **conditions** for approval are attached. The Signature page has been signed and returned with the **subject QAPP** to the RPM. Please return a copy of the completely signed signature page to FSS.

Attachment

cc: Steve Ostrodka, SMF-4J

ATTACHMENT

QUALITY ASSURANCE PROJECT PLAN (QAPP)
Amendment No. 2

The following comments should be addressed.

A Section 2.3.2, typo, Page 1 of 13
This should be Section 2.4.2 Project Objectives. See QAPP Page 9 of 17.

B Section 3.1, editorial
For this project the WESTON Site Manager is **Deepak L. Bhojwani**.

C ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SUMMA™ POLISHED CANISTERS BY GC/MS SELECTIVE ION MONITORING

❶ Page #5
Section 6.3 appears to be missing, or Section 6.0 is misnumbered.

❷ Section 9.2
Specify the concentrations of the 5 calibration levels.

❸ Appendix A Table A-1
The project required Compound **2-butanone** is not included in this table. See QAPP Table 4-4.

❹ Appendix A Table A-1
Provide Quantitation Ions for 2-butanone, surrogates, and internal standards.

❺ Appendix B
This page is blank. Are the TO-14 list of analytes supposed to be included. See Section 8.1.

❻ **AIR TOXICS LTD. (ATL) FAX** , Page 2
The project required Compound **Chloroform** is missing from this list. See QAPP Table 4-4.

❼ METHOD DETECTION LIMIT SUMMARY REPORT
The Method Detection Limits (MDLs) for the project required Compounds **acetone**, **methylene chloride, 2-butanone**, **freon 113**, and **chloroform** were not given. Can **ATL** achieve the project required MDLs as listed in Table A-1? See QAPP Table 4-4.

QUALITY ASSURANCE PROJECT PLAN

2.4.2 Project Objectives

The objective of the air sampling program is used to evaluate the concentrations of volatile organic compounds (VOCs) in the air, soil gas, and groundwater samples at the Evergreen Manor Site.

Air samples will be collected both inside and outside of the residences. Specifically, the following types of samples will be collected:

- Residential (Indoor air quality).
- Soil gas (Residential - Outside, near location of an indoor air sample).
- Ambient air (Residential- Outside, near location of a soil gas sample).
- Quality Control Samples (Field duplicates, equipment blanks).

The results of the sample analysis will be evaluated using the following the sequence of steps.

- *A qualitative comparison of the VOCs reported in air to those identified in the groundwater plume.*

  Based on the objectives of the sampling, the data analysis will focus only on those VOCs that have been identified by USEPA in the groundwater plume. Comparisons will account for the physicochemical properties of the VOCs of concern and their relative degradation products. Although, all chemicals that are analyzed and detected will be considered, the primary focus will be on chemicals that are present in both the groundwater and in the air samples.

- *A qualitative comparison of the VOC concentrations reported in residences to those reported in ambient air.*

  This evaluation will help establish a relationship between contaminants detected in the residences and those originating in the ambient air.

- *A qualitative comparison of the VOC concentrations in air reported in the residences*

to those reported in the soil gas.

This analysis will help determine if the VOCs from the plume are migrating through the soil and into basements.

- *A quantitative comparison of the VOC concentrations reported in air samples from the residences to risk based concentrations established by a recognized standard (e.g., risk-based concentrations developed by the United States Environmental Protection Agency Region III).*

  This evaluation will facilitate comparison of concentrations reported in the residential air samples with the accepted exposure limits and also assist in evaluating the level of risk associated with the reported concentrations.

Specific protocols associated with the air sampling activities are presented in the Field Sampling Plan (FSP) (Appendix A).

2.8 Project Schedule

A revised project schedule is shown in Figure 2-1b.

3.2.2 Validation of Analytical Data

Data validation of Special Analytical Services (SAS) air analyses data will be conducted by WESTON qualified data reviewers or a qualified data validation subcontractor. WESTON or the subcontractor will conduct the validation in accordance with the SAS request, method, laboratory SOPs, and National Functional Guidelines for Organic Data Review (Oct 1999), as applicable.

3.2.4 Scheduling of Special Analytical Services Analyses

WESTON's Sample Management Coordinator will coordinate all site sampling requirements with the WESTON procured an air laboratory.

3.2.5 Preparation of Special Analytical Services Requests

For all nonstandard sampling methods or parameters, a SAS request will be prepared by WESTON for approval by the U.S. EPA Region V FSS and/or U.S. EPA RPM/WAM. Each SAS request will contain specific methodology, analytical parameters, and detection limits. SAS requests have been prepared for the volatile air analysis. A copy of the SAS request is included in Appendix B.

3.4 LABORATORY OPERATIONS

Air analyses will be conducted by Air Toxics LTD in Folsom, California. Air Toxics LTD is a Weston procured SAS laboratory. WESTON has selected Air Toxics LTD to provide SAS analysis through a competitive bidding process.

Air Toxics Laboratory Director – Ms. Heidi Hayes is the laboratory director. Ms. Hayes is responsible for all overall technical performance of the laboratory. She is responsible for the quality of the analyses and the validity of the data.

Air Toxics Quality Assurance/Quality Control Officer (QA/QC) – Mr. James Parker supervises the quality of the data generated in the laboratory and the validation of the raw data generated. He is responsible for reporting the final data in accordance with the requirements of the contracts and projects. The QA/QC officer also reviews program plans for consistency with organizational and contractual requirements and will advise appropriate personnel of deficiencies. In addition, the QA/QC officer has the authority to stop work on projects if QC problems that affect the quality of the data produced arise.

Air Toxics Project Manager – Ms. Dede Dodge will be the project manager for the Evergreen Manor project. Ms. Dodge will be responsible for coordinating the samples with WESTON and the laboratory. Ms. Dodge will be the point of contact for any problems or concerns.

4.1 LEVEL OF QUALITY CONTROL EFFORT

Equipment blanks, field duplicates, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Equipment blank samples will be shipped to the laboratory as blind samples.

Equipment blank samples are analyzed to check for procedures at the site that may cause sample contamination. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are known as matrix spike/matrix spike duplicate (MS/MSD) samples.

The general level of the QC effort will be one field duplicate and one equipment blank for every ten or fewer investigative samples. The specific level of field QC for samples collected as part of the air study for the Evergreen Manor site is summarized in Table 2-1b of the FSP (Appendix A). Sampling procedures are specified in the FSP.

4.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSIS

The project required detection limits are presented in Table 4-4.

6.2.3 Transfer of Custody and Shipment Procedures

At the completion of sampling, samples will be packaged and shipped to the laboratory for analysis. The final vacuum will be noted on the chain of custody. This documentation will allow the lab to compare the vacuum from sampling with the receipt vacuum. The sample integrity is ensured if the final field reading and the lab receipt reading are similar. The sample may have been compromised during shipment, if the readings significantly differ. Custody seals will also be affixed across box entry points to provide another method of discerning if the samples were tampered with during shipment to the laboratory.

6.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

WESTON will either complete a SAS Packing List/Chain of Custody form or complete a COC using the FORMS II Lite Software.

6.3 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

The Air Toxics LTD SOP for sample receiving, login, and tracking of samples (SOP #50) is presented in Appendix G.

8.1 OFF-SITE LABORATORY ANALYTICAL PROCEDURES

Air samples will be analyzed for volatile organics by the air TO-15 SIM method (selective ion monitoring). Analysis will be conducted by the WESTON procured SAS laboratory. SUMMA canisters and air analysis will be provided by the laboratory. All SAS analysis will be in accordance with the protocols outlined in the respective SAS request in Appendix B. Analysis will be in accordance with the laboratory SOP #38 presented in Appendix G. Method detection limit studies,

initial calibration levels, and laboratory supplemental information to SOP #38, from the laboratory NELAP method manuals, is presented at the end of Appendix G.

10.2 LABORATORY SERVICES

10.2.1 Data Reduction

Data resulting from SAS analyses will be reduced, evaluated, and reported as described in the actual SAS request presented in Appendix B. Following data evaluation and reduction, the CLP-like data will be sent to WESTON or a WESTON data validation subcontractor for data validation.

10.2.2 Data Validation

WESTON, or the subcontractor, will conduct the validation in accordance with the SAS request, method, laboratory SOPs, and National Functional Guidelines for Organic Data Review (Oct 1999), as applicable.

The U.S. EPA Region V validation protocols for the data are based on the following guideline:

- *U.S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* —U.S. EPA, October 1999.

10.2.3 Data Reporting

The WESTON procured SAS laboratory will provide an electronic data deliverable (EDD)for the air analyses. The EDD will be in an excel format and EQUIS compatible. WESTON will utilize the laboratory generated EDD to assist in meeting the U.S. EPA Region V EDD required format for submittal on all superfund projects. WESTON will also incorporate field generated data into the EDD deliverable that will be submitted to U.S. EPA with the data summary report.

FIELD SAMPLING PLAN

2.7 AIR INVESTIGATION

Air sampling will be used to compare the concentrations of volatile organic compounds (VOCs) in the air, soil gas, and groundwater at the Evergreen Manor Site. The objective of the sampling event is to determine the relationship between the VOC-containing groundwater contaminant plume and the residential indoor air.

Samples will be collected both inside and outside of the residences. Specifically, the following types of samples will be collected:

- Residential (Indoor air quality).
- Soil gas (Residential - Outside, near location of indoor sample).
- Ambient air (Residential- Outside, near location of indoor and soil gas sample).
- Quality Control Samples (Field duplicates, equipment blanks).

Sampling will be performed for a twenty-four hour period at four residences. The canisters will be placed in the basement and first floor of the four selected residences. The sampling is designed so that all eight indoor air samples, sixteen soil gas samples, and 1 ambient air sample will be collected concurrently. In addition, 3 duplicate samples and 3 field/equipment blank will be obtained during the sample collection period for QA/QC. The duplicate samples will be collected in a separate SUMMA canister adjacent to the field sample.

3.8 AIR INVESTIGATION

3.8.1 Indoor Air Quality Sampling

Four residential air sampling locations will be chosen based on the proximity of the residences to monitoring well locations within the contaminated plume. Sampling locations are shown in Figure

2-3. In determining sample areas, WESTON reviewed historical groundwater data and the geology of the area to determine its impact on the pathway of the contaminated plume. Based on this assessment, it was determined that one residential home would be selected for air sampling in general areas surrounding each of the following monitoring wells:

- IEPA Well G104S,D and G105S, D (Area A)
- EPA MW-01 (Area B)
- EPA MW-02 (Area C)
- EPA MW-03 (Area D)

Residents in the above mentioned areas were contacted to determine their willingness to participate in the air sampling program and asked to complete a preliminary survey of their home. Some of the criteria for the selection of the resident sampling locations included:

- Proximity to the monitoring well.
- Configuration of the home.
- Other factors that would allow for an easy migration of vapors from the groundwater, through the soil and into a house.

Based on the survey, one residence from each area was chosen. Residences that were chosen for the air sampling study include:

- 4460 Mathew Ave., Roscoe, Illinois
- 4325 Straw Lane, Roscoe, Illinois
- 12007 Wagon Lane Ct., Roscoe, Illinois
- 11512 Wagon Lane, Roscoe, Illinois

Two indoor air quality samples, one from the basement and one from the first floor, will be collected from each residence. Using laboratory supplied SUMMA canisters, residential and ambient air samples will be collected as outlined in the steps below.

1. Fixed-rate flow controllers, along with 7-micron pre-filter attached to minimize entry of particulates, will be placed on the canister. Flow controllers will be set by the laboratory to meter the flow of air into the canisters at a relatively constant rate over a 24-hour sampling period. The fixed rate should allow the canisters to be filled to two-thirds of the capacity (a 4 liter sample for a 6 liter canister). Cleaned fixed flow controllers will be provided by the lab and used during the sample collection. Flow controllers will be used only once.

2. A vacuum gauge will be used to measure the initial and final vacuum of the canister, and to monitor the filling of the canister. The gauges will be used to provide a relative measure of change. Before sampling, the gauge will confirm the pressure reads between negative 29" and negative 30" Hg for each canister.

3. All residential (indoor) and ambient (outdoor) air samples will be collected at a uniform height during the sampling. To begin sampling, the flow controller will be attached to the sampler. All connections between the canister and the flow controller must be tight enough so that the various pieces of equipment (flow controller, gauge, etc.) when assembled cannot be rotated by hand. Any leaks in these connections will be corrected prior to sampling or the canister will be replaced. After the canister has been placed at the sample location, the canister inlet valve will be opened.

4. At the end of the sampling period (24 hours), the final vacuum for a canister will be measured using the vacuum gauge. The final canister vacuum should be between negative 4" and negative 12" Hg.

Appendix H presents a diagram depicting the equipment, which will be used to collect residential and ambient air samples. Sample information will be recorded on the SUMMA Canister Sampling Data Sheet provided in Appendix H. General meteorological conditions will be recorded during the sample period by downloading hourly weather observation data from the closest airport.

3.8.3 Soil Gas Sampling

Four soil gas samples will be collected at each of the four selected residences concurrently with the two indoor air samples. WESTON will utilize the U.S. EPA Geoprobe to collect samples from

depths 6 to 8 ft below ground surface (BGS) or below the foundation of the house, whichever is shallower. The SUMMA canister samples will be collected as outlined in the steps below.

1. WESTON will conduct utility clearance at the selected soil gas collection point. A metal detecting device, such as a "Metro Tech," will be used to provide additional subsurface utility clearance information.

2. The soil gas probe assembly and sampling will be completed by:

   a. Threading approximately 8 to 10 feet of ¼" thin-wall Teflon tubing through a 4 foot long, hollow stainless steel soil gas probe.

   b. Attaching a vented soil gas sampling tip to the Teflon tubing.

   c. Driving the soil gas probe assembly into the ground using a geoprobe as shown in Appendix H-Step 1. Add a second extension probe if necessary to sample at needed depth. Target depth is 6 to 8 feet below ground surface or below the foundation of the house, whichever is shallower.

   d. Removing the hollow probe, leaving the tip and Teflon tubing in the ground.

   e. Packing soil around the hole to seal the opening in the ground and connecting a brass Swage-loc 3/8" nut to the tubing outside the hole.

   f. Checking and recording the initial canister pressure (the reading should be approximately negative 30" Hg).

   g. Assembling the canister sampling train as indicated in Appendix H - Step 2 and in accordance with the following procedures. All sampling components are dedicated to each location.

      i. Attach the 24-hour critical flow orifice to the canister.
      ii. Connect the 7-micron particulate filter to the flow orifice.
      iii. Attach, using a brass 3/8" Swage-loc fitting, the Teflon tubing in the ground to the filter.
      iv. Attach the SUMMA canister to the Swage-loc fitting.
      v. Three volumes will be purged from the soil gas sampling probes.

3. When sampling is ready to begin, fully open the canister control valve will be opened fully.

4. At the end of the sampling period, the final vacuum for a canister will be measured using the vacuum gauge. The final canister vacuum should be between negative 4" and negative 12" Hg.

Sample information will be recorded on the SUMMA Canister Sampling Data Sheet provided in Appendix H. General meteorological conditions will be recorded during the sample period by downloading hourly weather observation data from the closest airport.

4.1 DUPLICATE SAMPLES

A duplicate sample will be collected to measure the agreement between canister samples. These samples will be collected using two (2) separate SUMMA canisters, critical flow orifices, and 0.7-micron filter connected with one (1) stainless steel T-fitting with a sampling cane. The samples will be collected using procedures as outlined in the previous sections. Opening and closing at the canisters will be the only additional step.

4.5 EQUIPMENT BLANKS

Equipment blanks will be collected to measure the potential contamination introduced by field sampling procedures, sampling media, sampling equipment, or shipment of the samples. The equipment blank will be collected using the following procedure:

1. Check the initial SUMMA canister pressure to verify that the vacuum is approximately negative 30" Hg. After this test, connect the critical flow orifice and 7-micron particulate pre-filter to the canister.

2. Ultra-high pure (UHP) nitrogen will be used as a zero air source and will be attached using a short piece of clean new Teflon tubing (less than 1 foot) to the sample tubing.

The nitrogen valve will be opened and the sample tubing flushed out. The canister sampling setup (canister and filter) connected by way of the sample tubing connected to the nitrogen and the canister valve will be opened. UHP nitrogen for blank samples will be obtained from the laboratory responsible for analyzing this project's air samples.

3. After the sample is collected, the canister valve will be shut and the filter removed.

5.1 PROJECT SAMPLE NUMBERING SYSTEM

Some examples of the WESTON project numbering system for the air sampling is as follows:

- EM2-AA01-B: Evergreen Manor site, phase 2, ambient air sample, basement sample (-F for first floor, -Y for Yard)
- EM2-GS01-N: Evergreen Manor site, phase 2, soil gas sample, sample collected on the North side of the house (-S for south, -W for West, and -E for east).
- EM2-AAFB01-01: Evergreen Manor site, phase 2, air sample, equipment blank sample.

5.2 SAS LABORATORY SAMPLE NUMBERING SYSTEM

Sample numbering for SAS numbers will follow the U.S. EPA Region V Central Regional Laboratory (CRL) sample numbering system. The CRL sample numbering system contains the following elements:

- 2002 - Indicates fiscal year (October 1, 2001 through September 30, 2002)
- ZG - Indicates Contractor Code (WESTON)
- Two-digit laboratory identifier number
- S - Indicates sample type (S = Sample, D = Duplicate, R = Blank)
- Two-digit sequential sample number (01-99)

An example SAS laboratory number is as follows: 2002ZG05S01 - The first sample collected by WESTON in fiscal year 2002 for the Evergreen Manor site for analysis by Air Toxics LTD.

6.3 SAMPLE DOCUMENTATION FORMS

SAS paperwork will consist of sample labels, sample tags, and SAS packing list/chain of custody forms or chain of custody forms generated by Forms II lite.

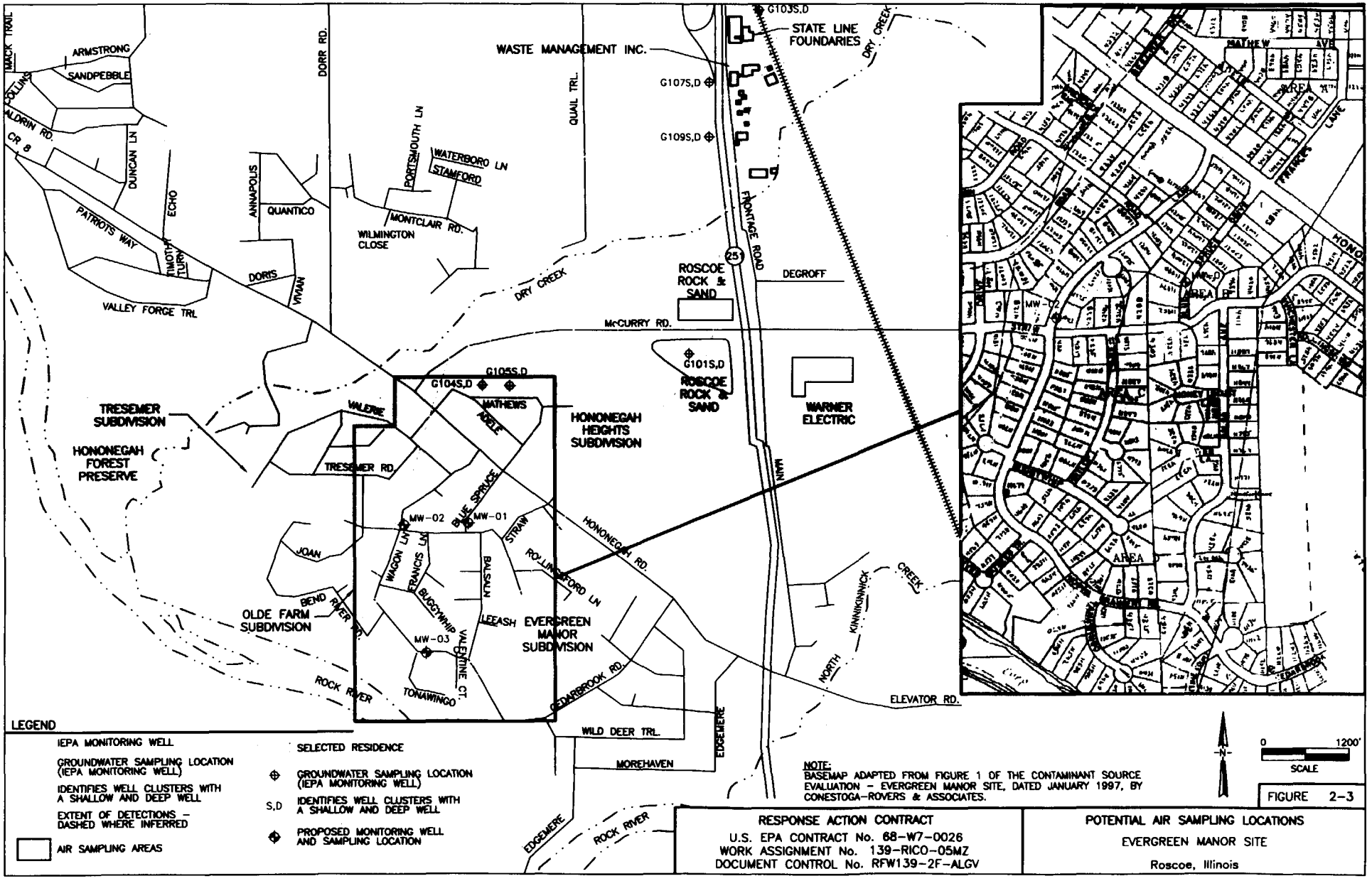
7.1 SAMPLE CONTAINERS AND SAMPLE PRESERVATION

Table 7-1b lists the required sample containers, sample volumes, sample preservation requirements, and holding times associated with the air analysis.

10.0 SAMPLE CONTAINER PROCUREMENT

All air samples (i.e. residential, soil gas, ambient air and quality control samples) will be collected using laboratory supplied SUMMA canisters. All canisters will be cleaned using the "SUMMA" process, which involves electro-polishing and chemical deactivation of the internal surface of the vessel using a combination of exponential dilution, heat and high vacuum. Each SUMMA canister will be certified by the laboratory as cleaned to the standards required for achieving the low sample detection limits. After cleaning, air from the canisters will be evacuated. The canisters will have a 6-liter capacity and a vacuum of approximately negative 30" Hg. SUMMA canisters for air analysis will be provided by Air Toxics LTD, the WESTON procured SAS laboratory. Preparation of SUMMA canisters will be accordance with the laboratory SOP #7 provided in Appendix G.

FIGURES


WASTE MANAGEMENT INC.
STATE LINE FOUNDARIES
G103S,D
G107S,D
G109S,D
DRY CREEK
QUAIL TRL.
FRONTAGE ROAD
ROSCOE ROCK & SAND
DEGROFF
McCURRY RD.
G101S,D
WARNER ELECTRIC
G105S,D
G104S,D
MATHEWS
ADELE
VALERIE
HONONEGAH HEIGHTS SUBDIVISION
TRESEMER SUBDIVISION
HONONEGAH FOREST PRESERVE
TRESEMER RD.
BLUE SPRUCE
MW-01
MW-02
MW-03
HONONEGAH RD.
ROLLINGFORD LN
STRAW
WAGON LN
FRANCIS LN
BUGGYWHIP
BALSAM
LEEASH
VALENTINE CT.
EVERGREEN MANOR SUBDIVISION
OLDE FARM SUBDIVISION
BEND RIVER RD.
JOAN
ROCK RIVER
TONAWINGO
CEDARBROOK RD.
WILD DEER TRL.
MOREHAVEN
EDGEMERE
ELEVATOR RD.
NORTH
KINNIKINNICK
CREEK
MAIN
ARMSTRONG
SANDPEBBLE
ALDRIN RD.
CR 8
DUNCAN LN
ECHO
PATRIOTS WAY
TIMOTHY TURN
VALLEY FORGE TRL
DORIS
VIVIAN
ANNAPOLIS
QUANTICO
DORR RD.
PORTSMOUTH LN
WATERBORO LN
STAMFORD
MONTCLAIR RD.
WILMINGTON CLOSE
MATHEW AVE
AREA A
AREA B
AREA C
AREA D
FRANCIS LANE
HONO
LEGEND
IEPA MONITORING WELL
GROUNDWATER SAMPLING LOCATION (IEPA MONITORING WELL)
IDENTIFIES WELL CLUSTERS WITH A SHALLOW AND DEEP WELL
EXTENT OF DETECTIONS - DASHED WHERE INFERRED
AIR SAMPLING AREAS
SELECTED RESIDENCE
GROUNDWATER SAMPLING LOCATION (IEPA MONITORING WELL)
S,D IDENTIFIES WELL CLUSTERS WITH A SHALLOW AND DEEP WELL
PROPOSED MONITORING WELL AND SAMPLING LOCATION
NOTE:
BASEMAP ADAPTED FROM FIGURE 1 OF THE CONTAMINANT SOURCE EVALUATION - EVERGREEN MANOR SITE, DATED JANUARY 1997, BY CONESTOGA-ROVERS & ASSOCIATES.
N
0
1200'
SCALE
FIGURE 2-3
RESPONSE ACTION CONTRACT
U.S. EPA CONTRACT No. 68-W7-0026
WORK ASSIGNMENT No. 139-RICO-05MZ
DOCUMENT CONTROL No. RFW139-2F-ALGV
POTENTIAL AIR SAMPLING LOCATIONS
EVERGREEN MANOR SITE
Roscoe, Illinois

TABLES

Table 4-4

Project Required Detection Limits Methods
Evergreen Manor
Roscoe, Illinois

Parameter	Air (ppbv)
Volatile Organic Compounds	
acetone	0.5
methylene chloride	0.1
2-butanone	0.1
cis-1,2-dichloroethene	0.02
1,1,1-trichloroethane	0.02
benzene	0.05
trichloroethene	0.02
toluene	0.05
tetrachloroethene	0.02
ethylbenzene	0.05
m & p xylene	0.05
o xylene	0.05
1,1,2-trichloro-1,2,2-trifluoroethane	0.02
chloroform	0.02

Table 2-1b

Summary of Sampling and Analysis Program for Evergreen Manor Roscoe, Illinois

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Equipment Blank			MS/MSD[1]			Matrix Total*
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq	Total	No.	Freq.	Total	
Air Samples - Residential	None	SAS Volatile Organics	8	1	8	1	1	1	1	1	1	1	1	1	10
Air Samples - Ambient	None	SAS Volatile Organics	1	1	1	-	-	-	-	-	-	-	-	-	1
Air Sample - Soil Gas	None	SAS Volatile Organics	16	1	16	2	1	2	2	1	2	-	-	-	20

Note: Three field blanks and three equipment blanks will be collected during the air analysis sampling. An MS/MSD does not count in the matrix total.

Table 7-1b

Sample Container, Volume, and Preservation Requirements
Evergreen Manor Site
Roscoe, Illinois

Matrix Type	Analysis	Sample Concentration Level	No. of Bottles	Type of Bottles	Preservatives	Technical Holding Time*
Air Samples (Residential, Ambient, and Soil Gas	Volatiles	Low	1	6 liter SUMMA Canister	N o preservation required	30 days

* All holding times are from the date of sample collection.

APPENDIX B

SAS

5/014-6/96

U.S. Environmental Protection Agency Region V
77 West Jackson, SMF-4J
Chicago, Illinois 60604
PHONE: (312) 886-1488 FAX: (312) 886-0753

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

A. EPA Region/Client:	Region V		
B. RSCC Representative:	H. Pham	Technical Project Officer (TPO): C. Moore	
C. Telephone Number:	(312) 353-2310	(312) 886-1488	
D. Date of Request:	March 2002	Date of Sampling : May 2002	
E. Site Name:	Evergreen Manor Roscoe, Illinois		
Cerclis ID#	ILD984836734	Site/Spill ID#	MZ

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

No deviations are allowed for any portion of the SAS without the permission WESTON.

A total of 31 air quality samples will be submitted for TO-15 SIM analysis. This includes:

- 8 indoor air samples
- 1 ambient/outdoor air samples
- 16 soil gas samples
- 3 field duplicate
- 3 equipment blank

The target compounds include:

- acetone
- methylene chloride
- 2-butanone
- cis-1,2-dichloroethene
- 1,1,1-trichloroethane
- benzene
- trichloroethene
- toluene
- tetrachloroethene
- ethylbenzene
- xylenes
- 1,1,2-trichloro-1,2,2-trifluoroethane
- chloroform

2. Definition and number of work units involved (specify whether whole samples or fractions; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 low concentration air samples will be submitted for TO-15 SIM analysis. Target compounds to include the list above.

3. Purposes of analysis (specify whether Superfund [Remedial or Enforcement], RCRA, NPDES, etc.):

Superfund Remedial

4. Estimated date(s) of collection:

May 2002 (tentatively scheduled for the week of May 20, 2002)

5. Estimated date(s) and method of shipment:

Daily, overnight courier (e.g.,Federal Express)

6. Number of days analysis and data required after laboratory receipt of samples:

The laboratory shall report analysis results within 14 days of receipt of last sample in shipment. A validatable, CLP-type data package is required.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Six-liter summa canisters to be supplied by the laboratory. Canisters will be individually-certified to S Levels, have 24-hr critical orifice settings, and include 0.7 micron pre-filter. Samples to undergo analysis by TO-15 SIM for target compounds.

Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Electronic data deliverable required.

QC REQUIREMENTS - Do not use designated field blanks or equipment blanks for QA audits.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The sample analysis data package shall be complete and shall include all documentation, data reporting forms and raw data for blanks, calibration verification standards, samples, duplicates, spike samples, laboratory control samples, dilutions, re-analyses, etc.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number and sample number.

10. Other (use additional sheets or attach supplementary information, as needed):

All original sample tags, chain of custody forms, SAS packing lists, airbills and any other original receiving or transmittal forms or copies of receiving logbook pages pertaining to this SAS shall be submitted within the time frame listed in section 6 above. Photocopies may be submitted along with a record of the location of the originals.

Payment to laboratories for this SAS analysis may be reduced if all procedures noted above are not followed and all required deliverables noted above are not supplied. The Region or its contractors shall not be charged further for the provision of required deliverables within this agreement.

Original data should be shipped to:

Tonya Balla
Roy F. Weston, Inc
750 E. Bunker Court
Vernon Hills, Illinois 60061
(847) 918-4055 (fax)

11. **Name of sampling/shipping contact:** Tonya Balla
Phone: (847) 918-4094

APPENDIX G

LABORATORY SOPS

Control Copy #:___

@A irT oxics Limited
STANDARD OPERATINGP ROCEDURE

PREPARATION OF SILCOSTEEL™ AND SUMMA™ CANISTERS FOR SAMPLING

SOP #7

RevisionDat e:	09/29/00
Revision#:	13
Reasonf or Revision:	ExternalUs e

Updated By:

Signature	Print	Title	Date

TechnicalR eview:

Signature	Print	Title	Date

QA Review:

Signature	Print	Title	Date

Laboratory Director:

LindaL. Freeman Date

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to outline the procedures for the cleaning and certification of 6 liters (6 L) and 1 liter (1 L) Silcosteel™ and Summa™ca nisters.

A. PURGINGANDEV ACUATION OF SUMMA™CAN ISTERS

2.0 EQUIPMENT/APPARATUS

- Custom stainless steel Purge Manifold able to purge and evacuate twelve canisterss imultaneously(Fig. 1)
- Charcoal(AgilentT echnologies)
- Zero Air Generator (Scott Specialty Gases, Plumsteadville, PA) catalog #53-42ZC)
- Dewar(PopeSci entific Inc., Menomonee Falls, WI) catalog #8640/0099
- SolenoidVal ve (Asco, SanM ateo,C A) catalog #8300A81
- Vacuumpum pca pable of10 $^{-4}$t orr
- GraLab Model 451 Digital Timer/Intervalometer (Dimco-Gray Co., Centerville, OH)

3.0 CLEANING OF CANISTERS

3.1 Prior to cleaning of canisters, the sample tags are removed and the date discarded recorded on the tag. Any dirt on the surface of the canister is removed.

3.2 PurgingP rocedure

Summa™ canisters are first purged for one hour with humidified high purity air. Canisters that have contained samples with concentrations exceeding approximately 10 ppmv are "red-tagged" by the analyst, and get an extra bake out to ensure cleanliness – see Section 3.3. Canisters are mounted up to twelve at a time onto a purge manifold. The system consists of automated filling and purging cycles. A minimum of twenty individual cycles are performed at 2 min intervals. A charcoal trap prevents vapors fromv enting intot he room. The total time is not critical, but rather the number of intervals must be a minimum of 20. Following this, the canistersar e evacuated.

3.3 High-Level (Non Sulfur) canisters – those marked with a "red tag" by the laboratory staff

3.3.1 These canisters are filled with humidified air following the last purge –s ee Section 3.2.

3.3.2 The pressurized canisters are capped with a 1/4" brass plug, the plastic canister valve knob is removed, and the canister is placed in ano ven.

3.3.3 The pressurized canisters are baked overnight (at least 8 hours) at 150°C.

3.3.4 The canistersar e purgedaga in – seeS ection3. 2.

The canisters are now ready for Thermal Cleanup and Final Evacuation.

3.4 Maintenance

3. 5.1 We ekly Maintenance

- The water level on the High Purity Air line is checked and replenished as needed. The source of water is the ion exchange system usedf or cleaninggl assware int heex traction lab.
- Ferrules arech angedas needed.
- The thermostat reading is recorded daily in each oven's logbook.

3.5.2 Monthly Maintenance

- The charcoal trap on the purge gas generator is replaced monthly.

4.0 THERMAL CLEANUP ANDFI NALEV ACUATION PROCEDURE

4.1 Threeca nisters areco nditioneds imultaneously.
4.2 Place a Dewaro f liquid Argon(2/3t o 3/4f ull)un der theco ld trap.
4.3 Turno n ConvectronGauge (power strip).
4.4 Seto venr acksf or theapp ropriate size canisters.
4.5 Connectt he canisterst ot hepur ge manifold.
4.6 Turn on the oven and set the temperature for ≥125°C (oven setting noted on the oven).
4.7 Turno n thepum p.

4.8 Record the **date, canisters' serial numbers, time, oven run #,** and the technician's **initials** in the logbook. Refer to *Appendix A* for an example of an oven logbook page.

Note: The oven temperatures are verified on a daily basis. As is noted in Section 3.5.3, the thermostat reading is recorded in each oven's logbook.

4.9 **Leak check as follows:** with the valves closed and pump on, ensure that the gauge goes below 100 mTorr within one minute. If this occurs, there are no leaks in the canister valves. If this does not occur, the leaking canister is determined and removed from the system for repair.

4.10 Open the valves on the canisters slowly, one at a time.

4.11 Close the oven door.

4.12 The technician notes the time in the logbook and the canisters are heated for a minimum of one hour for 6L canisters and 1/2 hour for 1 L canisters.

4.13 After at least one hour, turn off the heat, open the oven doors, note the time in the logbook, and verify that the pressure is less than 20 mTorr.

4.14 When the canisters have cooled (approximately 20 min), verify that the vacuum is ≤10 mTorr, and record the **final vacuum** in the logbook.

4.15 Close the valves on the canisters.

4.16 Turn off the pump.

4.17 Disconnect the canisters from the purge manifold.

4.18 Place a brass plug on each canister.

4.19 Attach a yellow and a blue silicone gasket on the canister.

4.20 Stamp the date cleaned on the back of the yellow tag for canisters to be submitted for certification and record which oven was used.

6.0 CLEANING OF COLD TRAP

Occasionally canisters contain liquids such as water. This liquid accumulates in the cold trap upon evacuation of the canister, and its presence is obvious. It is essential that this liquid be removed. An inventory of clean cold traps is kept on hand so that the time necessary for this procedure does not interfere with production. The process for removing this liquid is as follows:

6.1 Immerse the cold trap in liquid Argon (which freezes the liquids and prevents odors) and remove the trap from the system.

6.2 Remove the trap from the liquid Argon and immediately move the trap to a fume hood.

6.3 In the hood, rinse the cold trap several times with methanol and deionized water. Place these rinses in the organic waste container.

6.4 The final rinse should be methanol.

6.5 Place the clean trap in a canister-cleaning oven to dry. The trap is ready for use after 2- 3 hours in the oven.

7.0 CERTIFICATION

7.1 Ten percent of the canisters cleaned are selected randomly and certified quarterlyb yGC /MS analysisf orT O-14t argetco mpounds.

7.2 Canisters are analyzed in sets of three as they are removed from the cleaningo vens. T he ovenus ed. date canisters were cleaned. batch number. and individual canister numbers are recorded on the Weekly Canister Certification Report form(*AppendixB*).

7.3 Canisters are analyzed by GCMS TO-15. Canisters that pass are recleaned via the thermal cleanup station only, and then placed in the shipment inventory after a canister certification sticker has been affixed to the yellow tag. Thepas sing criteria aren oted in Table 1 below. Canisters that fail are recleaned priort or eanalysispe r theb atchce rtificationpr otocol.

Table 1. Passing criteria

Can Size	Cleaning Level	Standard TO-14 Compounds	Polar/Nonstandard TO-14Com pounds SIM	Polar/Nonstandard TO-14 Compounds
6Li ter	Low Level	0.1ppb v	ProjectS pecific	0.5ppb v
6 Liter	Normal	0.2 ppbv	NA	0.8ppb v
1 Liter	Normal	2.0 ppbv	NA	2.0ppb v

7.4 A summary of canister certifications, that tallies the number of passes and fails, is provided to the QA department on a monthly basis. A copy of this summaryi sal sokept i n theca nister cleaningr oom.

7.5 The canisters submitted for certification are tracked in the canister logbook using thef ollowing codes:

X = DoubleP urge (highl evel canister)
C = Canister tob ece rtified
P = Passed Certification (also highlighted with a yellow highlighting pen for easyr eference)
FC = Failed Certification,r ecertify
XC = High level canistert o be certified- was double purged

8.0 CERTIFICATION OF SUMMA CANISTERSA ND ASSOCIATED FLOW CONTROLLERS

8.1 Materials

- UHP N2 Tankwi thReg ulator

- StainlessS teelM anifold capable ofat taching 56L summa canisters
- Flow Controllers
- Cleaned 6L summa canisters

8.2 Procedure

8.2.1 Open the UHP N2 regulator to a flow rate of > 30 ml/min. through them anifold.
8.2.2 While the N2 is purging the manifold attach the summa canisters andf low controller assemblies.
8.2.3 Open the canistersv alves
8.2.4 When theca nisters reach 5ps i closet he canisterv alves
8.2.5 Remove canisters fromt he manifold and attachb rass caps.
8.2.6 Theca nisters aren ow readyt o be certified.

9.0 SHIPPING PROCEDURES

9.1 Materials

- Thickwa lledwh itei ndividualc anister box(appropriates ize)
- Thin walledwh ite external shipping box
- BubbleP ack
- "AirT oxics" White/BlueBo xLa bel
- Laboratory "Chain-of-Custody"f orm

9.2 Procedure

9.2.1 Placeea ch canisteri nto a relativelyt ight fittingi ndividualb ox.
9.2.2 Placef our canister boxes inside one outer white shippingb ox.
9.2.3 Include a "Chain-of-Custody"f orm in eachb ox.
9.2.4 Include a Certificate of Cleanliness (*Appendix C*) noting the batch certification inea chb ox.
9.2.5 Fill out any necessary forms for shipping and attach them to the shipping box.

Listof Appendices
Appendix A. Exampleof an Oven Logbook Page
Appendix B WeeklyC anister CertificationR eport
AppendixC Exampleof aC ertificationof Cleanliness

AppendixA

Example ofan Ov enL ogbookPage

OvenC @Air Toxics Ltd LogBo ok # 507

Date	Set	Start Time	End Time	Can	Code	Can	Code	Can	Code	End mTorr	Init.	Comments.	Temp.: (°C)

Page 1 Signature Date Revised 5/99

Appendix B

Weekly Canister Certification Report

Weekly Canister Certification Report — Week of ____________

Oven	Date	Canister Number	Fail	Fail	Fail	Pass	6L	1L	Silco	TO-14/SIM Short List/Low Level
Initials	Batch									
Notes can# 1:										
Notes can# 2:										
Notes can# 3:										

Oven	Date	Canister Number	Fail	Fail	Fail	Pass	6L	1L	Silco	TO-14/SIM Short List/Low Level
Initials	Batch									
Notes can# 1:										
Notes can# 2:										
Notes can# 3:										

Oven	Date	Canister Number	Fail	Fail	Fail	Pass	6L	1L	Silco	TO-14/SIM Short List/Low Level
Initials	Batch									
Notes can# 1:										
Notes can# 2:										
Notes can# 3:										

Oven	Date	Canister Number	Fail	Fail	Fail	Pass	6L	1L	Silco	TO-14/SIM Short List/Low Level
Initials	Batch									
Notes can# 1:										
Notes can# 2:										
Notes can# 3:										

Oven	Date	Canister Number	Fail	Fail	Fail	Pass	6L	1L	Silco	TO-14/SIM Short List/Low Level
Initials	Batch									
Notes can# 1:										
Notes can# 2:										
Notes can# 3:										

Totals for this page=

Totals for this week=

Sum total of canisters certified this week=

Number of canisters cleaned this week=

Percent Cleaned=

Percent Passed =

O:Files/Excel/Other/Canclean/cancert%.xls Revised 6/13/00

Appendix C

Example of a Certification of Cleanliness

Controlled Copy #:___

@ AIR TOXICS LIMITED
STANDARD OPERATING PROCEDURE

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SUMMA™ POLISHED CANISTERS BY GC/MS SELECTIVE ION MONITORING

EPA METHOD TO-14/TO-14A/TO-15

SOP #38

Revision Date: 10/22/99
Revision #: 2
Reason for Revision: External Use

	Signature	Print	Title	Date
Updated By:				
Technical Review:				
QA Review:				
Laboratory Director:		Linda L. Freeman		

1.0 SCOPE AND APPLICATION

To provide a procedural guide for the application of EPA Method TO-14 to the analysis of low level volatile organic compounds in ambient air using evacuated stainless steel summa canister collection. To describe specific adaptations of the method to the analysis of the EPA Clean Air Act List of target compounds.

2.0 METHOD SUMMARY

2.1 Description

EPA Method TO-14 describes techriques for the analysis of airborne VOCs collected as whole air samples in stainless steel canisters. 500 mL of air is withdrawn from the canister through a mass flow controller and cryofocused at -189°C in a dewar of liquid argon. The focused air sample is then flash heated through a hydrophobic drying system which removes water from the sample stream prior to analysis by GC/MS in the SIM mode.

2.2 Deviations

Modifications to EPA Methods TO-14/TO-14A/TO-15 used by ATL to carry out the analyses of air samples are summarized in Table 1 (page 2).

3.0 HEALTH AND SAFETY

Normal laboratory safety precautions must be used when preparing standards from neat materials for this method and when analyzing samples. These precautions include working in a fume hood, wearing eye protection, and wearing a laboratory coat when handling neat materials. Also, precautions must be taken to avoid skin contact with neat materials. Refer to the compound specific MSDS for additional information. Care must also be taken when handling syringes to ensure that a needle stick does not occur.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

An air sample is collected in an evacuated stainless steel Summa canister or Tedlar bag. Upon receipt, the canisters will be between ca. 10" Hg vacuum and ambient pressure. Canister receipt vacuum/pressure is reported on the first page of the data report. If abnormal conditions exist the project contact is notified by the sample custodian or a client service representative before analysis can begin. Data from such samples will be qualified. Prior to analysis, the canister is pressurized to 5 psig for 6 L canisters and 15 psig for 1 L canisters. Samples are stored in the sample cage in the main laboratory. Analysis must occur within 3 days for Tedlar bags and 30 days for canisters.

Table 1. Summary of Method Modifications

Requirement	TO-14	TO-14A	TO-15	ATL Modifications
Sampling/concentrator system	Nafion Drier	Nafion Drier	Multisorbent concentrator	Multisorbent concentrator
Canister cleaning - clean air supply	Cryogenic Trap	Cryogenic Trap	Cryogenic Trap	Use of Humidified UHP Air
Canister certification	Pressurize w/humidified zero air.	Pressurize w/humidified zero air.	Pressurize w/humidified zero air.	Pressurize w/dry UHP nitrogen
Sample load volume	400 mL	400 mL	Not mandated	Up to 0.5 liter
Blank	Humid air blank	Humid air blank	Humid air blank	Humid air blank for standard analysis. Dry air blank for low level analysis.
Blank acceptance criteria	< 0.2 ppbv	< 0.2 ppbv	< DL	< DL
BFB absolute abundance criteria	Within 10% of that from previous day.	Within 10% of that from previous day.	Not addressed	CCV surrogate recoveries demonstrate stability from one day to the next
BFB acceptance criteria	SW-846 Protocol	SW-846 Protocol	CLP protocol	SW-846 protocol
Concentration of IS spike	Not specified	Not specified	10 ppbv	25 ppbv for TO-14; 10 ppbv for low level
Dilutions for initial calibration	Dynamic dilutions or static using canisters.	Dynamic dilutions or static using canisters.	Dynamic dilutions or static using canisters.	Syringe dilutions
Flow rates/operating parameters				Optimixed. See procedures section.
ICAL RRF %RSD acceptance criteria	Not specified	Not specified	30% or less, 40% or less for up to two compounds	30% or less for standard compounds, 40% or less for non-standard and polar compounds
IS recoveries	Not specified	Not specified	Within 40% of mean over ICAL for blanks, and w/in 40% of daily CCV for samples.	Within 40% of CCV recoveries for blank and samples.
IS RTs	Not specified	Not specified	Within .33 min from most recent calibration (either ICAL or daily)	Within 0.5 min. of RT in daily CCV
Daily CCV	Not specified	Not specified	70 - 130%	Standard compounds: 70 – 130% for at least 90%; Non-standard and polar compounds: 60 - 140% for at least 80%
RF for quantitation	From ICAL	From ICAL	From daily CCV	From ICAL
Canister leak check	24 hour, positive pressure	24 hour, positive pressure	24 hour, positive pressure	20 minute, vacuum check
MSD scan range	18 - 250 amu	18 – 250	35 - 300 amu	35 – 350 amu

		amu		

5.0 *INTERFERENCES AND POTENTIAL PROBLEMS*

Interferences to this method generally include high levels of carbon dioxide, water and/or heavy hydrocarbons. High levels of CO_2 in the samples can cause freezing of the trap and thus a flow drop on the sample interface. When this occurs, a smaller amount of sample is collected resulting in higher dilution factors. Very high levels of moisture in the samples cause erratic internal standard and surrogate responses and therefore likely erratic target compound responses. When a sample has high levels of heavy hydrocarbons, the analyst may have to dilute the sample more than the target compound level requires, ensuring the system is not contaminated.

6.0 *EQUIPMENT/APPARATUS*

6.1 List of Equipment/Apparatus

- 25 µL, 500 µL, 1 mL, 2.5 mL, 5 mL, 25 mL, & 50 mL gastight syringes (CMS)
- Certified NIST Traceable VOC blends - Scott Specialty Gases
- Aldrich Chemical High Purity Neat Standards
- Tedlar Bags (1L, 3L and 10L) SKC
- Heating Tapes - Various Lengths (Cole Palmer)
- Power Controllers (Cole Palmer)
- HP 5973 MSDs with (UNIX Operating System) Thru-Put Software
- NIST /NBS75.1K Library Search Software
- DB-624 105 m X 0.53 Megabore column
- Liquid Argon 5500 cu. ft. dewar (Local Supplier)
- Ultra High Purity Helium (Local Supplier)
- Liquid cryogenic dewars (300 mL, 500 mL, 5000 mL) CMS
- Laboratory designed cyrofocusing TO-14 interface equipped for sampling and backflushing contents of the cryotrap to the drier. The cryotrap consists of 1/8" stainless steel tubing packed with acid washed glass beads and wrapped around a cartridge heater. Purge/desorption gas flow is regulated by a flow controller on the canister interface. Optional syringe injection of gaseous standards is accomplished through a septum cap just prior to the sampling valve.
- Laboratory designed drying system consists of a hydrophobic sorbent housed in a heated block. Water passes through the drier at ambient temperature while organic VOCs are retained. Following the drying cycle, the tube is heated to 250°C and the VOCs are backflushed to the GC/MS through a heated transfer line connected to the packed column injection port.
- Tylan Mass Flow Control Module (1 to 100 mL/min. air)
- Edwards Vacuum Pump

- Laboratory Designed Heated Dynamic Gas Dilution Manifold - based on flow controllers and digital flow sensors for calibration and diluent gases
- Laboratory Designed Canister Receiving Station equipped with high-resolution vacuum/pressure gauge and diluent gas inlet.

6.2 Analysis is carried out on a GC/MS system equipped with a Megabore™ inlet adapter, cryogenic oven controller, a J&W Scientific DB-624 column and a Hewlett-Packard 5973 Mass Selective Detectors.

6.4 SIM parameters

Both the primary and secondary ions, for each target compound are, selected for monitoring by GC/MS software. Compounds are identified using both the ion profiles generated and the retention time windows.

6.5 Quantitation is based on the internal standard technique. The internal standards, bromochloromethane, chlorobenzene-d_5 and 1,4-difluorobenzene are spiked into each standard, blank, sample and QC sample at 10 ppbv. The surrogates 1,2-dichloroethane-d_4, toluene-d_8 and 4-bromofluorobenzene are also spiked into all standards, blanks, samples and QC samples at 10 ppbv.

7.0 CANISTER MOUNTING AND SPIKING PROCEDURES

7.1 Upon receipt, the canister vacuum/pressure is recorded. Canister receipt vacuum/pressure is reported on the front page of the data report. If abnormal conditions exist the project contact is notified by the sample custodian or a client services representative before analysis can begin. Data from such samples will be qualified. Prior to analysis, the canister is pressurized to 5 psig for 6 L canisters and 15 psig for 1 L canisters.

7.2 Canisters are connected to the inlet line of the cryofocusing unit. Connections are leak checked by monitoring the flow. As vacuum is achieved, the flow will drop to zero. After leak checking is complete, the valve on the canister is opened and flow allowed to equilibrate. During this time, a 1 mL injection of IS/Surrogates is made.

7.3 Sampling is initiated by rotating the valve into the sample position. Sampling continues for a period of 10 minutes or until the desired volume of air has been withdrawn.

8.0 STANDARD PREPARATION

8.1 Stock Standards

Standards are prepared from NIST Traceable Scott Specialty Gas VOC blends and supplemented with neat materials. The standards are blended into the working range by taking known aliquots and using density-based calculations. Maximum standard holding times are noted below. In addition, if standard degradation is observed (e.g., poor recoveries), new standards are prepared.

The original TO-14 list of analytes* is purchased from Scott Specialty Gases as a 5 ppmv in a high pressure cylinder blend *(Appendix B)*. The

blend is produced by the manufacturer using criteria designed to minimize vapor phase interactions and maximize long-term stability.

** Excluding benzyl chloride, styrene, and both trans- & cis-1,3-dichloropropene, which are included in the neat materials mix, Section*

Inject 480 mL of the 5 ppmv certified TO-14 standard into an evacuated 6 L canister that contains 50 µL of water. This canister is then used for the combined standard described in Section 8.3.

8.2 Neat Materials Calibration Blend

Analytes not present in the Scott Gas blends are purchased in neat form. These compounds are blended in the gas phase as follows. A Tedlar bag is filled with an accurately measured volume of UHP N_2. Prescribed amounts of the neat material are then injected into the bag. Density based calculations are used to determine the prescribed amounts and final concentrations.

Note: Tedlar bags are used for the static dilution medium due to their inherent inertness to polar analytes. Standards should not, however, be stored in the Tedlar bags beyond 3 days. Fresh calibration standards are prepared and then transferred to Summa canisters for storage. The standards prepared from neat materials are stable in Summa canisters for 6 months.

Appropriate amounts of neat materials are spiked into a Tedlar bag filled with UHP nitrogen. The standard is then drawn into an evacuated 6 L Summa canister and then pressurized. The final concentration of the standard is 50 ppmv.

8.3 Combined Calibration Blend

For the combined standard, 48 mL of the neat laboratory blended standards described in Sections 8.2 are added to the certified TO-14 standard canister described in Section 8.1. After bringing the canister to 15 psi (a 1:2 dilution), the final concentration of all analytes is 200 ppbv. Aliquots are then introduced onto the instrument by mass flow controller. Standards prepared in this manner may be used for up to 3 month from date of preparation.

8.4 Internal Standard/Surrogate Mix

Blend the prescribed amount of neat material into a Tedlar bag to a final volume of 10 L. Transfer the contents of the Tedlar bag to an evacuated Summa canister and pressurize to 15 psig (1:2.01 dilution). The canister is then vented to 0 psig a second time and re-pressurized to 15 psig (1:2.01 dilution). The canister is vented to 0 psig a third time and re-pressurized to 20 psig (1:2.36 dilution) for a final concentration of 5 ppmv. This type of standard may be used for up to 6 months.

	COMPOUND	DENSITY	FW	µL	ppmv
	Bromochloromethane	1.991	129.39	1.3	48.1
	Chlorobenzene-d_5	1.157	117.60	2.0	47.3
	1,4-Difluorobenzene	1.110	114.09	2.0	46.8
(Surrogate)	1,2-Dichloroethane-d_4	1.666	236.06	2.8	47.5
(Surrogate)	Toluene-d_8	0.943	100.21	2.1	47.5
(Surrogate)	4-Bromofluorobenzene	1.593	175.01	2.2	48.2

One mL of the IS blend is injected into the canister interface as each standard blank, and sample is being loaded. The IS amount loaded in this fashion will be 10 ppbv per component.

8.5 BFB Check Mix

Two µL of a certified, 99% pure 25 ng/µL 4-bromofluorobenzene standard purchased from Supelco is injected for the 50 ng on-column tune check. The standard is stored in sealed ampules and expires 1 year after the opening date, or the manufacturer's expiration date, whichever comes first.

9.0 CALIBRATION AND QUALITY CONTROL PROCEDURES

The following sections discuss method-specific quality control procedures. Generic control limits are used for evaluation of TO-14 instrument performance. Laboratory limits are established for surrogate recoveries as a second means for evaluating the analytical systems.

9.1 BFB Tune Check

A daily (every 24-hours, or every 12-hours if project requires) tune check with 4-bromofluorobenzene is achieved by directly injecting 2 µL of the BFB check standard into the GC in accordance with SW-846 tuning criteria in the full scan mode. The relative abundances of selected ions are tabulated and reported as outlined in Appendix C. Analysis cannot proceed unless all criteria of the tune check are met.

9.2 Initial Calibration

The percent relative standard deviations (%RSD) for all standard TO-14 compounds must be ≤30%, and for all non-standard and polar compounds, must be ≤40%. Otherwise a new initial calibration curve is performed. In those instances where the RSD for one or more analytes exceeds 30%, the average of the RSD values is determined by summing the RSD value for each analyte and dividing by the total number of analytes. Calibration curve is considered acceptable if the mean RSD for all analytes in the calibration is ≤ 30%.

The multipoint calibration is constructed by loading varying amounts of the combined calibration blend (Section 8.3) on the canister interface. The standard volume of air withdrawn is 0.5 liter, consequently lower loadings result in an effective dilution.

Calibration is typically performed using five levels, though certain projects only require three. One standard must be at or near the reporting limit.

The Relative Response Factor (RRF) from the daily continuing calibration verification (mid-level standard) is used to quantitate results unless a project specifically requires that the average RRF from the initial calibration curve is used.

9.3 Second Source Calibration Verification

A second source check containing all target compounds is analyzed after each initial curve, to verify that the standards are correct and the accuracy of the calibration. The acceptance criterion for the second source recoveries is as follows. For the "Standard" TO-14 list compounds, recoveries for 90% of the compounds must be ±30%. For the "Non-standard and Polar" TO-14 list compounds recoveries for 80% of the compounds must be ±40%. The delineation between standard and non-standard TO-14 compounds can be found in Appendix A. (Some projects require a ±25% second source recovery criterion for 100% of the target compounds.) If the noted limits are exceeded, the problem is investigated and if warranted, a new analytical curve is analyzed for the out-of-limits compound(s).

9.4 Continuing Calibration Verification

A continuing calibration verification (CCV) is performed at the start of each day and if required by a specific project, every 12 hours. This is an analysis of the primary source mid-level calibration standard. The acceptance criteria for the percent difference (%D) between the daily CCV

response and average response from the calibration curve is as follows. For the "Standard" TO-14 list compounds, the %D for 90% of the compounds must be ≤30%. For the "Non-standard and Polar" TO-14 list compounds the %D for 80% of the compounds must be ≤40%. (*See Appendix A* for the delineation between standard and non-standard TO-14 compounds.) If the CCV fails to meet the performance criteria then maintenance should be performed and the test repeated. If the system still fails the calibration check, a new 5-point calibration curve is performed.

Certain projects have different CCV acceptance criteria (e.g., ≤25% for 100% of compounds. A specific list of target analytes is requested when the CCV acceptance criteria are required differ than the ATL standard criteria noted above.

9.5 Laboratory Control Sample (LCS)

When required for specific projects, a mid-level spike using the second source standard is analyzed daily prior to sample analysis. The acceptance criteria are identical to those noted in Section 9.4. If the stated criteria are not met, the system is checked and the standard re-analyzed. In the event the criteria cannot be met, the instrument is re-calibrated.

9.6 Laboratory Blanks

A moist system blank then a dry laboratory blank is run after the CCV at the beginning of each day and at least once in every 24 hour shift. A laboratory blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist in the chromatographic system. The acceptance criterion for laboratory blanks is a result less than the laboratory reporting limit *(Appendix A)*.

9.7 Internal Standards

The internal standards retention times for the blanks, QC samples and samples must be within ±0.5 minutes (30 seconds) of the retention times in the continuing calibration check. In addition, the internal standard area must be within ±40% of the CCV's internal standard area for all blanks, QC samples, and samples. A warning limit of ±30% is used to investigate possible misinjection of the internal standard. When samples are analyzed on the same 24-hour clock as the initial calibration curve, the mid-point standard will be used for evaluation of internal standards.

If the internal standards for the blank do not pass the acceptance criteria, the system is inspected and the blank reanalyzed. Analyses do not continue until the blank meets the internal standard criteria.

If the internal standard acceptance criterion is not met, the sample must be analyzed again unless obvious matrix interference is documented. If the criterion is still not met, then the data is reported from the first analysis and the matrix effect narrated in the laboratory narrative included with the data report. Upon request, the data from the matrix effect confirmation analysis is provided to the client.

9.8 Surrogates

The acceptance limits for surrogate recoveries are 70 to 130%. If the surrogate recoveries for the blank are outside of these limits, the system is inspected and the blank reanalyzed. Analysis does not continue until the blank meets the surrogate recovery criteria. If the recovery limits are not met for sample analysis, the sample must be re-analyzed unless obvious matrix interference is documented. If the limits are still not met, then the data from the first analysis is reported and the out-of-limit surrogate recovery flagged.

9.9 Laboratory Duplicates

A duplicate sample analysis is performed on 10% of the samples. The relative percent difference (RPD) between the two analyses must be $\leq 25\%$ for all compounds detected at greater than 5 times the reporting limit. If this limit is exceeded, the sample is re-analyzed a third time. If the limit is exceeded again, the cause is investigated and the system brought back to working order. If no problem is found on the system, the data is flagged to note the non-conforming event.

9.10 End Checks

Certain projects require that the CCV be repeated at the end of the analytical sequence. The acceptance criterion is $\pm 30\%$ of the expected value, except for certain projects that require recoveries of 15%. If a compound fails, the system is checked and the standard re-analyzed. If the 2nd analysis of the end check fails, the system is considered to be out of control. After the source of the problem is identified and necessary corrective action is performed, a subset of the affected day's samples are reanalyzed to determine at what point the analytical system began to fail. Reanalysis is performed where necessary.

9.11 Method Detection Limit Studies

Method Detection Limit (MDL) studies are analyzed as described in 40 CFR Pt. 136 App. B. The MDL determines the 99% confidence level for seven replicates of a low-level standard. The MDL is defined as 3.14 times the standard deviation of the seven replicates and must be less than the

reporting limit. Ability to accurately detect and quantitate target compounds at the reporting limit is verified by a running reporting limit-level standard each time a multi-point calibration curve is analyzed. See *Appendix A* for the TO-14 by SIM mode reporting limits.

10.0 CALCULATIONS

$$\text{Relative Response Factor (RRF)} = \frac{\text{Area of Compound}}{\text{Area of Int. Standard}} \times \frac{\text{Conc. Int. Standard (ppbv)}}{\text{Conc. of Compound (ppbv)}}$$

$$\underset{\text{(ppbv on-column)}}{\text{Results Calculation}} = \frac{\text{Area of Compound in Sample}}{\text{Area of Int. Standard in Sample}} \times \frac{\text{Conc. Int. Standard (ppbv)}}{\text{ICAL RRF*}}$$

CCV: Continuing Calibration Verification

** The average RRF from the CCV curve is used as required by specific projects.*

ppbv in sample = ppbv on-column X dilution factor

Note: The dilution factor includes canister pressurization dilution and any subsequent dilution required to ensure all results are within the instrument calibration range.

11.0 SAMPLE ANALYSIS

11.1 Analytical Batch

The analytical batch is defined as the number of samples analyzed in one analytical day. The number of samples analyzed in one analytical day varies depending on the number of samples received for a particular project, the age of the samples upon receipt, the sample holding times (three days for Tedlar bag samples and 30* days for Summa canister samples), and the project-specific QC requirements. A maximum of 12 samples/analytical batch are analyzed when 12-hour tune-clocks are required.

**Some projects require a 14-day holding time for canister samples.*

Samples are logged into a work order as noted on the chain-of-custody. As many samples on one work order as physically possible are analyzed within one analytical batch. If samples extend to more than one analytical batch, appropriate QC will be reported.

11.2 Analytical Sequence

Initial 24-hour period	*Subsequent 24-hour period*
BFB Tune Check	BFB Tune Check
Initial Calibration	CCV
Reporting Limit Verification	LCS - if required by project
Independent Calibration Check	Laboratory Blank
Laboratory Blank	Samples
Samples	End Check - if required
End Check - if required	

The "Subsequent 24-hour" sequence is followed each 24 hour period (every 12 hours when specified by the project) that samples are analyzed, until the system is found to be out of calibration.

11.3 Sample Dilutions

To obtain analyte concentrations within the linear range of the detectors and to prevent contamination of the system, samples are screened prior to analysis on a GC/FID. The total VOC concentrations are approximated and used to determine the volume of sample loaded for TO-14 analysis. A 1:1 dilution involves loading 500 mL of the sample. Most dilutions are performed by loading a lesser sample volume. The dilution factor is obtained by dividing 500 mL by the sample volume loaded. This technique is also used for calibration. Alternatively, samples that contain over approximately 20.000 ppmv of target analytes are diluted by adding a measured aliquot of the sample to a Tedlar bag that has a metered volume of nitrogen as the diluent gas*. For this alternative dilution technique, up to 500 mL of the diluted sample is loaded. As noted in Section 5.0, the final dilution factor includes canister pressurization dilution as well.

** This concentration is based on a 1:1000 bag dilution, then a subsequent 1:500 dilution to an approximate on-column concentration of 40 ppbv for a 1 mL load.*

11.4 Qualitative Target Compound Identification

Compounds are qualitatively identified based on retention time and by comparing background subtracted sample spectra to the reference library spectra. An analyte is qualitatively identified when the following two criteria are met:

- the Relative Retention Time (RRT) for the analyte must be within ±0.06 RRT units of the RRT of the analyte in the daily continuing calibration check;

- ions present in the standard spectrum greater than 10% of the most abundant ion must be present. Also, the relative intensity of the ions greater than 10%, must be ±20% of the intensity in the standard spectrum.

The ion intensity test is performed by the GC/MS software. Ions that do not meet the intensity criteria are flagged in the raw data. Failure to meet the intensity criteria may be indicative of matrix interference or low signal to noise (i.e., low concentration).

It may be necessary for the analyst to perform manual background subtraction of coeluting peaks to obtain a clean spectrum. The analyst must always use his/her discretion on any identification.

12.0 CORRECTIVE ACTION PROCEDURES

A request for corrective action (CAR) is initiated any time either the ATL SOPs or client-prescribed QC protocol are not followed, or in any other instance that sample results are adversely affected. The corrective action is documented in ATL's Corrective Action Procedures SOP #61.

13.0 DATA REVIEW

13.1 Initial Data Review

As the analytical sequence is run throughout the day, the data is reviewed by the bench chemist using the following eight steps:

13.1.1 Check for any project-specific requirements.
13.1.2 Verify holding time.
13.1.3 Verify the tuning criteria, CCV, LCS (when applicable), and end check (when applicable)
13.1.4 Verify method blank has no hits above detection limit.
13.1.5 Verify sample results.
  a) Verify internal standards, retention time, and surrogates recoveries are acceptable.
  b) Verify correct amount of sample analyzed.
  c) Verify the reasonableness of the automated peak integration and spectral verification.
  d) Verify result concentrations are within linear range of calibration curve (upper 50% for dilutions.)
13.1.6 Initial and date raw data and or logbook entry to indicate that the data is acceptable.
13.1.7 Apply data appropriate data flags.
13.1.8 Describe unusual events on data review sheet.

Notes:

- *Preparation and review of laboratory narrative are carried out as explained in the SOP # 45.*
- *Compilation of data package by the analyst/scientist is carried out as described in the data review SOP #22.*

13.2 Secondary Data Review

The team leader or a QA-approved peer performs a secondary technical data review on 100% reports. This review follows all the steps mentioned in the initial data review (Section 13.1).

13.3 Tertiary (QA) Data Review

A thorough tertiary data review is performed by the QA department on a randomly chosen 10% of the final data packages. The QA review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a corrective action report and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and detection limits are also checked. Field and trip blanks are checked and trends are observed.

13.4 Quaternary Data Review

Some clients requests that 100% of their final data packages undergo a fourth technical review. The quaternary reviews in this case are performed by the team leader, QA-approved peer, or QA personnel.

14.0 INSTRUMENT MAINTENANCE

Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real time feedback to the analyst on the condition of the instruments. Routine maintenance includes:

14.1 Mass Spectrometers

- Daily check of vacuum ion gauge (Increase in ion count indicates a potential leak)
- Daily (every 24 hours) autotune check with BFB
- Cleaning of ion source on quarterly basis or as needed
- The oil level and quality is visually checked on a monthly basis to insure proper vacuum pump function, and oil is changed every 6 months.

14.2 Sample Introduction System

The mass flow controller used for sample introduction is sent for off-site calibration against NIST-certifiable source once every two years.

To ensure a clean sample introduction system, if necessary, the lines and trap are "steam-cleaned" by analyzing a humidified system blank. This takes place every day following standards (i.e., CCV) analysis. Humidified system blanks are also analyzed after saturation-level detections in samples.

14.3 Gas Chromatograph

Basic maintenance includes the following: (*Every 6 months or more frequently if needed)*

14.3.1 Clip 3 feet off the front end of the capillary column, and if necessary, the backend as well.

14.3.2 Replace the injection port liner. The liner is replaced by removing the inlet cap using a wrench and releasing the liner from the inlet body using a pair of tweezers. Care should be taken not to get fingerprints on any inside surface.

14.3.3 Once per week change septa on the GC and once per day change the septa on the valve syringe interface (injection port). Always use Supelco Thermogreen septa and take care not to leave fingerprints on any inside heated surface. Wear a pair of white cotton gloves or use tweezers to handle the septa. Lower the oven temperature to 40°C. Remove the inlet cap with a wrench, remove the old septa with a pair of tweezers and insert the new septa.

14.3.4 The column is replaced when chromatography peak shape or resolution degrades. Similarly, if the column bleed profile rises with age then the column needs replacing. Use new black graphite ferrules each time and clip off approximately 1" of column after inserting it through the ferrule. This will remove any graphite particles that may have scrapped off into the column. Tighten the column nut and ferrule finger tight and one-quarter turn with a wrench. Tightening any more only crushes the ferrule and may damage the column.

14.3.5 The bench analyst will document any routine or major maintenance in the bound instrument logbook assigned to each instrument. The date of the maintenance, what work was performed and analyst initials are included.

15.0 DELIVERABLES

Data reporting packages are prepared as described in SOP# 41 – Preparation of Hardcopy Analytical Reports Using ADT.

16.0 REFERENCES

EPA Method TO-14
Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Revision 1 June, 1988.

EPA Method TO-14A
Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1997. *EPA/625/R-96/010b*

EPA Method TO-15
Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1997. *EPA/625/R-96/010b*

SW-846 Method 8000B
Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, Revision 1, December, 1996.

Volatile Organic Analysis of Ambient Air in Canisters - *Draft Method*
USEPA Contract Laboratory Program, Revision VCAA01.0, December 1991

List of Appendices

Appendix A. Reporting Limits & Quantitation Ions
Table A-1 Reproting Limits & Quantitaion Ions for Standard TO-14 List
Table A-2 Reporting Limits and Quantitation Ions for Non-standard TO-14 List
Appendix B. Scott Specialty Gas Blend Certificates of Analysis
Appendix C. BFB Tune Criteria & Check Mix

Appendix A

Table A-1. Reporting Limits & Quantitation Ions for Standard TO-14 List

Compound	Reporting Limit (ppbv)	Quantitation Ion	Qualifier Ion
Freon 12	0.02	85	87
Freon 114	0.02	135	137
Chloromethane	0.02	50	52
Vinyl Chloride	0.010	62	64
Bromomethane	0.02	96	94
Chloroethane	0.02	64	66
Freon 11	0.02	101	103
1,1-Dicloroethene	0.010	61	96
Freon 113	0.02	151	153
Methylene Chloride	0.05	49	94
1,1-Dichloroethane	0.02	63	65
cis-1,2-Dichloroethene	0.02	61	96
Chloroform	0.02	83	85
1,1,1-Trichloroethane	0.02	97	99
Carbon Tetrachloride	0.02	119	117
Benzene	0.05	78	77
1,2-Dichloroethane	0.02	62	64
Trichloroethene	0.02	95	130
1,2-Dichloropropane	0.02	63	62
cis-1,2-Dichloropropene	0.02	75	77
Toluene	0.02	91	92
trans-1,2-Dichloropropene	0.02	75	77
1,1,2-Trichloroethane	0.02	97	99
Tetrachloroethene	0.02	166	129
1,2-Dibromoethane	0.02	107	109
Chlorobenzene	0.02	112	114
Ethyl Benzene	0.02	91	106
m,p-Xylene	0.02	91	106
o-Xylene	0.02	91	106
Styrene	0.02	104	78
1,1,2,2-Tetrachloroethane	0.02	83	85
1,3,5-Trimethylbenzene	0.02	105	120
1,2,4-Trimethylbenzene	0.02	105	120
1,3-Dichlorobenzene	0.02	146	148
1,4-Dichlorobenzene	0.02	146	148
1,2-Dichlorobenzene	0.02	146	148
1,2,4-Trichlorobenzene	0.02	180	182
Hexachlorobutadiene	0.05	225	223

Appendix A

Table A-2. Reporting Limits & Quantitation Ions for Non-Standard TO-14 List

Compound	Reporting Limit (ppbv)	Quantitation Ion	Qualifier Ion
1,3-Butadiene	0.10	54	39
Acetone	0.10	43	58
trans-1,2-Dichloroethene	0.10	61	96
Bromodichloromethane	0.10	83	85
Dibromochloromethane	0.10	129	127
Bromoform	0.10	173	171

Appendix B

Scott Specialty Gas Blend Certificates of Analysis

8.0 TO-14/TO-15 – VOLATILE ORGANIC COMPOUNDS

This method involves full scan and Selective Ion Monitoring (SIM) GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14/15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and is either cryofocused via liquid Argon or concentrated using a multisorbent bed. The focused air sample is then flash heated through a hydrophobic drying system which removes water from the sample stream prior to analysis by full scan GC/MS or by GC/MS in the (SIM) mode.

For low level analysis, a cryogenic valve is employed to cold trap the gases onto the GC column.

ATL performs a modified version of this method. The summary of method modifications, standard target analyte list, Reporting Limits, QC criteria, and QC summary can be found in the following tables.

Table 8-1. Summary of Method Modifications

Requirement	TO-14	TO-14A	TO-15	ATL Modifications
Sampling/Concentrator System	Nafion Drier	Nafion Drier	Multisorbent concentrator	Multisorbent concentrator
Canister Cleaning - Clean Air Supply	Cryogenic Trap	Cryogenic Trap	Cryogenic Trap	Use compressed air
Canister Certification	Pressurize with humidified zero air.	Pressurize with humidified zero air.	Pressurize with humidified zero air.	Pressurize with dry UHP Nitrogen
Sample Load Volume	400 mL	400 mL	Not mandated	Up to 0.5 liter
Blank	Humid air Blank	Humid air Blank	Humid air Blank	Humid Nitrogen Blank for standard analysis. Dry Nitrogen Blank for Low Level/SIM analysis.
Blank Acceptance Criteria	< 0.2 ppbv	< 0.2 ppbv	< RL	< RL
BFB Acceptance Criteria	SW-846 Protocol	SW-846 Protocol	CLP protocol	SW-846 protocol
Concentration of IS Spike	Not specified	Not specified	10 ppbv	25 ppbv for TO-14; 10 ppbv for Low Level/SIM analysis
Dilutions for Initial Calibration	Dynamic dilutions or static using canisters	Dynamic dilutions or static using canisters	Dynamic dilutions or static using canisters	Syringe dilutions
ICAL RRF %RSD Acceptance Criteria	Not specified	Not specified	30 % or less, 40 % or less for up to two compounds	30 % or less for standard compounds, 40 % or less for non-standard and polar compounds or ≤ 30 % pooled RSD of all compounds

Continued

Table 8-1. Summary of Method Modifications – *Continued*

Requirement	TO-14	TO-14A	TO-15	ATL Modifications
IS Recoveries	Not specified	Not specified	Within 40 % of mean over ICAL for blanks, and w/in 40 % of daily CCV for samples.	Within 40 % of CCV recoveries for blank and samples.
IS RTs	Not specified	Not specified	± 0.33 min from most recent calibration (either ICAL or daily)	± 0.33 min of RT in daily CCV
Daily CCV	Not specified	Not specified	70 – 130 % R	Standard compounds: 70 – 130 % for at least 90%; Non-standard and polar compounds: 60 – 140 % for at least 80 %
RF for Quantitation	From ICAL	From ICAL	From daily CCV	From ICAL
Canister Leak Check	24 hour, positive pressure	24 hour, positive pressure	24 hour, positive pressure	Vacuum check prior to analysis
MSD Scan Range	18 - 250 amu	18 – 250 amu	35 - 300 amu	35 - 350 amu

Table 8-2. Method TO-14/TO-15 Analyte List (Standard Compounds)

Analyte	RL for SIM / Low Level / Standard / (ppbv)	Low Point of Curve for SIM / Low Level / Standard / (ppbv)	Acceptance Criteria	
			ICAL/LCS/CCV Accuracy (% R)	Precision (% RPD)
1,1,2,2-Tetrachloroethane	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,1,2-Trichloroethane	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,1-Dichloroethane	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,1-Dichloroethene	0.01 / 0.1 / 0.5	0.01 / 0.1 / 0.5	70 – 130	≤ 25
1,2,4-Trichlorobenzene	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	70 – 130	≤ 25
1,2,4-Trimethylbenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,2-Dibromoethane (Ethylene Dibromide)	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,2-Dichlorobenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,2-Dichloroethane	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,2-Dichloropropane	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,3,5-Trimethylbenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,3-Dichlorobenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
1,4-Dichlorobenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Benzene	0.05 / 0.1 / 0.5	0.05 / 0.1 / 0.5	70 – 130	≤ 25
Bromomethane	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Carbon Tetrachloride	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Chlorobenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Chloroethane	0.05 / 0.1 / 0.5	0.05 / 0.1 / 0.5	70 – 130	≤ 25
Chloroform	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Chloromethane	0.05 / 0.1 / 0.5	0.05 / 0.1 / 0.5	70 – 130	≤ 25
Chlorotoluene (Benzyl Chloride)	0.1 / 0.1 / 0.5	0.1 / 0.1 / 0.5	70 – 130	≤ 25
cis-1,2-Dichloroethene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
cis-1,3-Dichloropropene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Dichloromethane (Methylene Chloride)	0.1 / 0.1 / 0.5	0.1 / 0.1 / 0.5	70 – 130	≤ 25
Ethylbenzene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Freon 114	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Hexachlorobutadiene	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	70 – 130	≤ 25
m,p-Xylene	0.04 / 0.1 / 0.5	0.04 / 0.1 / 0.5	70 – 130	≤ 25
Methyl Chloroform (1,1,1-Trichloroethane)	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
o-Xylene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Styrene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Tetrachloroethene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Toluene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
trans-1,3-Dichloropropene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Trichloroethene	0.02 / 0.1 / 0.5	0.02 / 0.1 / 0.5	70 – 130	≤ 25
Vinyl Chloride	0.01 / 0.1 / 0.5	0.01 / 0.1 / 0.5	70 – 130	≤ 25

Table 8-3. Method TO-14/TO-15 Analyte List (Non-Standard and Polar Compounds)

Analyte	RL for: SIM / Low Level / Standard / (ppbv)	Low Point of The Curve for SIM / Low Level / Standard / (ppbv)	Acceptance Criteria	
			ICAL/LCS/CCV Accuracy (% R)	Precision (% RPD)
1,3-Butadiene	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	60 – 140	≤ 25
1,4-Dioxane	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
2-Butanone (Methyl Ethyl Ketone)	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	60 – 140	≤ 25
2-Hexanone	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
4-Ethyltoluene	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	60 – 140	≤ 25
4-Methyl-2-Pentanone (MIBK)	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Acetone	0.5 / 0.5 / 2.0	0.5 / 0.5 / 2.0	60 – 140	≤ 25
Bromodichloromethane	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Bromoform	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Carbon Disulfide	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Cyclohexane	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Dibromochloromethane	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Ethanol	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Heptane	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Hexane	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Isopropanol	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Methy t-Butyl Ether (MTBE)	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	60 – 140	≤ 25
Propylene	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
Tetrahydrofuran	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
trans-1,2-Dichloroethene	0.1 / 0.5 / 2.0	0.1 / 0.5 / 2.0	60 – 140	≤ 25
Vinyl Acetate	NA / 0.5 / 2.0	NA / 0.5 / 2.0	60 – 140	≤ 25
TPH or NMOC (Hexane/Heptane)	NA/2.0/10	1 pt. calibration	NA	≤ 25

Table 8-4. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 - 140
1,4-Difluorobenzene	60 - 140
Chlorobenzene-d_5	60 - 140

Table 8-5. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 8-6. Summary of Calibration and QC Procedures for Methods TO-14/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires	SW - 846 tune criteria	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis	% RSD ≤ 30 for all "standard" compounds and % RSD ≤ 40 for all "non-standard" Compounds	Correct problem then repeat Initial Calibration Curve.
LCS	After each Initial Calibration Curve and daily prior to sample analysis	Recoveries for 90 % of "standard" compounds must be 70 - 130 %; for 80 % of "non-standard" compounds recoveries must be 60 - 140 %	Investigate the problem and if warranted, analyze a new analytical curve for the out-of-limits compounds.
Reporting Limit Verification (RLV)	With each Initial Calibration	Within documented historical control limits for 90 % of "standard" compounds and for 80 % of the "Non-standard" compounds	Check the system and re-analyze the Standard. Notify QA Dept. via a CAR form if fail to meet the criteria
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours	% D ± 30 for 90 % of "standard" compounds; % D ± 40 for 80 % of the "non-standard" compounds	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5-point calibration curve.
Laboratory Blank	After the CCV	Results less than the laboratory Reporting Limit	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention Time (RT) for the blanks and samples must be within ± 0.33 minutes of the RT in the CCV. The IS area must be within ± 40 % of the CCV's IS area for the blanks and samples	**For blanks:** inspect the system and re-analyze the blank. **For samples:** re-analyze the sample unless obvious matrix interference is documented. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out of limits a second time, report data from the first analysis and narrate.
Surrogates	As each standard, blank, and sample is being loaded	70 – 130 % R	**For blanks:** inspect the system and re-analyze the blank. **For samples:** re-analyze sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %R are out-of-limits a second time, report data from the first analysis and narrate.
Laboratory Duplicates	10% of the samples	RPD ≤ 25 % for detections > 5 X's the RL	Re-analyze the sample a third time. If the limit is exceeded again, Investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

eport Date : 13-Aug-2001 10:21 Page 1

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

ethod File: /chem/msdw.i/w-11auga.b/to14810fmdl.m
atch File: /chem/msdw.i/w-11auga.b
ast ID: msdw.i

TO-14 S/M

ID:	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07
LENAME:	w081110	w081111	w081112	w081113	w081114	w081115	w081116
J.DATE:	11-AUG-2001	11-AUG-2001	11-AUG-2001	11-AUG-2001	11-AUG-2001	11-AUG-2001	11-AUG-2001
J.TIME:	16:48	17:43	18:20	19:06	19:45	20:24	21:12

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL
1 Propylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
2 Dichlorodifluoromethan	43.99	46.26	45.00	43.14	45.94	45.14	46.68	45.16	1.27	3.98
3 Freon 114	46.69	45.46	45.57	43.95	44.82	46.36	44.53	45.35	0.97	3.06
4 Freon 22	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
5 Chloromethane	56.65	54.36	56.41	52.61	54.04	53.11	50.88	54.01	2.06	6.47
6 Vinyl Chloride	49.73	46.41	48.60	49.65	50.09	51.84	48.43	49.25	1.68	5.26
7 1,3-Butadiene	245.65	237.91	244.06	244.54	238.85	252.06	252.42	245.07	5.69	17.89
8 Dimethyl Ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
9 Bromomethane	31.13	26.22	29.08	27.23	28.67	26.52	18.91	26.82	3.88	12.20
10 Chloroethane	43.57	47.59	42.90	48.06	45.43	49.08	42.55	45.60	2.67	8.40
11 Trichlorofluoromethane	47.95	49.01	48.84	57.35	48.26	48.62	48.38	49.74	3.37	10.60
12 Freon 113	49.39	50.02	48.76	48.66	46.92	47.90	46.87	48.36	1.19	3.76
13 Acrolein	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
14 1,1-Dichloroethene	50.62	50.84	50.07	49.02	49.77	50.55	49.10	50.00	0.73	2.30
15 Acetone	391.08	363.20	370.56	356.24	369.96	374.60	362.62	369.75	11.22	35.26
16 Ethanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
17 2-Propanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++

conc. = ppbv

Loaded 100mL of #197-34
TO14 → 0.25ppbv
AT → 1.25ppbv

Final conc. TO14 → 0.05 ppbv
AT → 0.20 ppbv
Based on 500mL loads

Reviewer 1 DP Date: 8-13-01
Reviewer 2 [illegible] Date: 8/24/01

WL 8-24-01

APR.-30 02:17PM 10:22 AIRTOXICS LTD

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msdw.i/w-11auga.b/to14810fmdl.m
Batch File: /chem/msdw.i/w-11auga.b
Inst ID: msdw.i

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL
*	40 1,4-Difluorobenzene	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	0.00	0.00
	41 Trichloroethene	49.60	44.24	46.60	45.27	46.73	48.54	46.14	46.73	1.83	5.77
	42 1,2-Dichloropropane	45.86	44.73	47.69	45.93	45.98	47.88	46.76	46.40	1.12	3.51
	43 1,4-Dioxane	252.20	234.95	245.43	235.98	239.88	250.94	240.79	242.88	6.86	21.56
	44 Bromodichloromethane	221.16	219.07	220.04	214.28	224.58	228.54	220.02	219.81	6.14	19.31
	45 cis-1,3-Dichloropropen	53.14	49.96	51.04	48.70	50.70	50.32	49.76	50.52	1.38	4.33
	46 4-Methyl-2-pentanone	226.15	223.71	225.42	219.92	221.17	226.89	215.25	222.64	4.15	13.04
$	47 Toluene-d8	10.58	9.94	10.56	10.24	10.92	10.96	10.83	10.58	0.38	1.18
	48 Toluene	47.63	43.28	46.84	45.87	49.32	49.99	48.37	47.33	2.27	7.15
	49 trans-1,3-Dichloroprop	44.65	45.56	43.48	43.95	40.94	42.40	40.41	43.06	1.90	5.97
	50 1,1,2-Trichloroethane	43.54	45.04	43.23	44.89	42.29	42.85	41.22	43.29	1.36	4.29
	51 2-Hexanone	208.78	215.05	209.27	212.94	199.67	199.98	190.67	205.34	8.91	27.99
	52 Tetrachloroethene	42.64	45.31	41.55	43.88	40.90	40.78	40.34	42.23	1.90	5.96
	53 Dibromochloromethane	192.88	205.93	192.04	197.27	181.80	185.19	180.82	190.85	9.03	28.37
	54 1,2-Dibromoethane	40.13	49.50	46.71	46.35	45.46	45.28	43.35	45.26	2.93	9.19
*	55 Chlorobenzene-d5	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	0.00	0.00
	56 Ethyl Benzene	46.24	43.82	45.87	44.76	44.63	47.86	43.62	45.26	1.50	4.71
	57 Chlorobenzene	47.74	46.10	46.67	45.87	46.20	47.01	45.64	46.46	0.73	2.30
	58 m,p-Xylene	92.18	90.68	91.34	91.02	89.96	91.46	89.47	90.87	0.92	2.90
	59 o-Xylene	46.17	43.19	44.69	43.60	44.04	50.06	42.72	44.92	2.53	7.95
	60 Styrene	58.86	51.79	57.32	57.45	56.10	56.44	54.85	56.12	2.28	7.16
	61 Bromoform	187.40	195.29	182.15	195.43	169.83	172.87	167.30	180.04	10.31	32.41
$	62 Bromofluorobenzene	10.20	9.83	10.36	10.20	10.59	10.78	10.59	10.36	0.32	1.01
	63 1,1,2,2-Tetrachloroeth	51.72	51.96	49.15	50.32	47.83	49.45	47.66	49.73	1.71	5.37

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msdw.i/w-11auga.b/to14810fmdl.m
Batch File: /chem/msdw.i/w-11auga.b
Inst ID: msdw.i

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL
64 4-Ethyltoluene	228.32	223.50	224.97	224.18	220.29	215.83	220.18	222.45	4.04	12.71
65 1,3,5-Trimethylbenzene	54.37	44.60	44.95	48.80	46.37	44.06	46.05	47.03	3.59	11.30
66 Octane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
67 1,2,4-Trimethylbenzene	50.91	45.03	42.98	43.79	44.43	42.83	43.40	44.76	2.82	8.86
68 1,3-Dichlorobenzene	59.51	51.54	53.80	52.59	54.38	53.73	52.17	53.96	2.65	8.32
69 1,4-Dichlorobenzene	59.50	49.54	50.89	52.20	52.23	50.77	49.91	52.15	3.40	10.69
70 Benzyl Chloride	35.68	31.05	33.62	31.64	32.27	33.23	32.06	32.77	1.53	4.81
71 1,2-Dichlorobenzene	67.92	53.48	57.63	55.89	56.93	57.31	57.49	58.09	4.57	14.37
72 1,3-Dichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
73 Dibromomethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
74 1,2,4-Trichlorobenzene	108.86	80.12	80.70	85.44	76.43	76.73	68.77	82.44	12.72	39.98
75 Hexachlorobutadiene	85.22	72.15	71.64	67.67	69.30	69.43	68.31	71.96	6.07	19.08
76 Naphthalene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++

Report Date : 19-Oct-2001 08:51

TO-14 SIM full List

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /var/chem/msdx.i/x-18oct.b/to14110f.m
Batch File: /chem/msdx.i/x-18oct.b
Inst ID: msdx.i

ID:	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07
FILENAME:	x101806	x101807	x101808	x101809	x101811	x101813	x101815
INJ.DATE:	18-OCT-2001	18-OCT-2001	18-OCT-2001	18-OCT-2001	18-OCT-2001	18-OCT-2001	18-OCT-2001
INJ.TIME:	15:01	16:13	16:53	17:38	18:18	19:06	19:52

(PPTV) RL

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL	
1 Propylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
2 Dichlorodifluoromethan	53.52	50.32	53.15	53.27	59.15	55.27	52.35	53.60	2.92	9.14	20
3 Freon 114	52.94	50.77	56.15	54.72	57.97	57.87	51.22	54.52	2.98	9.37	20
4 Freon 22	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
5 Chloromethane	52.62	51.30	55.36	56.49	60.57	56.35	54.92	55.37	2.95	9.39	20
6 Vinyl Chloride	46.08	42.13	46.72	40.80	40.07	43.06	40.22	42.73	2.73	8.58	10
7 1,3-Butadiene	144.20	143.41	146.84	142.40	157.71	155.56	142.46	147.51	6.44	20.23	100
8 Dimethyl Ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
9 Bromomethane	11.45	10.20	10.62	12.95	10.73	18.43	15.62	12.86	3.09	9.70	20
10 Chloroethane	48.66	50.39	56.39	49.13	54.71	54.35	46.48	51.41	3.68	11.57	50
11 Trichlorofluoromethane	39.40	36.89	40.49	51.35	51.44	42.07	38.31	42.85	6.06	19.06	20
12 Freon 113	54.54	54.44	56.68	55.00	60.40	58.17	53.78	56.15	2.41	7.58	20
13 Acrolein	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
14 1,1-Dichloroethene	51.40	50.10	53.74	51.34	58.25	57.85	52.06	53.53	3.27	10.28	10
15 Acetone	181.12	161.90	166.43	162.25	175.46	180.11	166.69	170.56	8.19	25.75	500
16 Ethanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
17 2-Propanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

loading 100 ml of # E55-46
TO-14 → 0.25ppbv
Al. extra → 0.625ppbv

Based on 500 ml load the on column concentration is
TO-14 0.05ppbv
Al. extra 0.125ppbv

Reviewer 1 Levi Ford Date: 10-19-01
Reviewer 2 W. Brand Date: 10/22/01

10.19.01

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /var/chem/msdx.i/x-18oct.b/to14110f.m
Batch File: /chem/msdx.i/x-18oct.b
Inst ID: msdx.i

(PPTV) RL

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL	
18	Carbon Disulfide	120.86	118.67	124.79	123.41	134.35	133.72	119.12	124.99	6.55	20.60	100
19	1-Pentene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
20	Pentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
21	Methylene Chloride	98.46	83.58	68.36	88.57	95.38	93.53	86.32	90.60	5.32	16.73	100
22	MTBE	125.96	123.61	131.38	127.55	142.25	142.02	134.40	132.43	7.50	23.56	100
23	trans-1,2-Dichloroethe	125.36	121.24	129.74	123.97	138.64	136.08	126.38	128.74	6.39	20.09	100
24	Hexane	150.77	220.62	129.73	125.96	142.69	138.70	128.52	133.86	10.57	33.23	100
25	1,1-Dichloroethene	49.52	48 56	50.47	46.40	52.16	50.59	46.91	49.23	2.08	6.52	20
26	Vinyl Acetate	96.64	99.24	103.54	99.22	109.53	114.71	105.18	104.01	6.41	20.15	100
27	2-Butanone	101.83	101.06	105.81	101.57	112.83	115.84	104.58	106.22	5.87	18.45	100
28	cis-1,2-Dichloroethene	50.81	49.61	54.29	51.89	55.67	55.72	52.90	52.98	2.37	7.45	20
* 29	Bromochloromethane	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	0.00	0.00	
30	Tetrahydrofuran	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
31	Chloroform	55.52	53.01	56.00	53.86	60.15	59.52	54.13	56.03	2.80	8.79	20
32	Cyclohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
33	1,1,1-Trichloroethane	53.89	51.83	56.55	52.36	58.93	57.11	52.87	54.79	2.73	8.59	20
34	Carbon Tetrachloride	18.67	17.59	19.62	17.80	17.08	20.64	17.11	18.36	1.35	4.25	20
$ 35	1,2-Dichloroethane-d4	9791.30	9666.98	10362.40	9729.75	10235.66	10470.26	9696.25	9993.23	348.20	1094.39	
36	1-Hexene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
37	Benzene	58.84	54.78	54.46	55.62	56.91	56 21	56.75	56.22	1.48	4.66	50
38	Heptane	135.80	126.95	122.31	128.18	126.22	131.31	130.50	128.75	4.30	13.50	100
39	1,2-Dichloroethane	61.10	60.25	59.38	59.86	60.06	59.31	59.96	59.99	0.60	1.88	20

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /var/chem/msdx.i/x-18oct.b/to14110f.m
Batch File: /chem/msdx.i/x-18oct.b
Inst ID: msdx.i

(PPTV) RL

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL	
*	40 1,4-Difluorobenzene	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	0.00	0.00	
	41 Trichloroethene	57.06	56.59	55.43	57.95	58.41	57.10	57.08	57.09	0.96	3.01	20
	42 1,2-Dichloropropane	53.55	52.72	52.37	53.61	54.18	56.72	53.43	53.80	1.42	4.47	20
	43 1,4-Dioxane	131.09	129.16	127.29	128.57	138.37	136.95	135.20	132.38	4.42	13.89	100
	44 Bromodichloromethane	113.14	114.28	113.16	113.72	116.39	114.16	113.18	116.01	1.15	3.63	100
	45 cis-1,3-Dichloropropen	49.77	50.38	47.07	49.30	51.42	52.02	51.62	50.23	1.72	5.39	20
	46 4-Methyl-2-pentanone	104.15	103.60	102.66	106.80	104.44	108.86	103.45	104.85	2.19	6.89	100
$	47 Toluene-d8	10079.50	10101.85	9924.07	10090.73	9654.59	10136.26	10052.84	10005.69	168.88	530.80	20 within LR
	48 Toluene	55.18	55.30	52.28	54.72	54.06	56.67	54.82	54.72	1.34	4.20	20
	49 trans-1,3-Dichloroprop	52.58	52.91	53.67	50.99	57.49	53.88	53.80	53.62	1.98	6.23	20
	50 1,1,2-Trichloroethane	55.00	55.89	56.56	56.01	61.12	54.90	56.52	56 57	2.21	6.64	20
	51 2-Hexanone	103.57	98.06	102.73	103.06	102.55	104.51	96.22	101.53	3.33	9.78	100
	52 Tetrachloroethene	57.60	57.46	57.59	58.20	62.74	57.65	58.45	58.53	1.89	5.95	20
	53 Dibromochloromethane	114.42	114.10	116.29	114.39	126.97	113.37	113.14	116.30	4.90	15.41	100
	54 1,2-Dibromoethane	52.59	57.89	58.85	52.77	57.61	52.23	58.14	55.73	3.02	9.48	20
*	55 Chlorobenzene-d5	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	10000.00	0.00	0.00	
	56 Ethyl Benzene	61.32	59.27	56.99	54.90	54.66	56.87	59.98	57.71	2.55	8.02	20
	57 Chlorobenzene	57.73	56.63	55.85	57.43	58.86	57.21	56.59	57.21	0.95	2.98	20
	58 m,p-Xylene	126.08	114.09	111.03	109.01	109.95	114.72	118.78	114.81	5.98	18.81	40
	59 o-Xylene	50.23	49.09	47.52	48.98	49.44	51.40	50.48	49.59	1.25	3.94	20
	60 Styrene	50.32	48.59	53.53	48.45	48.10	51.93	55.73	50.95	2.91	9.15	20
	61 Bromoform	100.98	99.72	101.23	100.44	110.88	101.42	105.29	102.85	3.96	12.45	100
$	62 Bromofluorobenzene	9844.51	9725.25	9656.28	9326.94	9056.21	9656.02	9708.89	9567.73	275.87	867.05	
	63 1,1,2,2-Tetrachloroeth	53.80	53.32	52.07	53.00	57.09	53.06	51.84	53.42	1.75	5.49	20

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /var/chem/msdx.i/x-18oct.b/to14110f.m
Batch File: /chem/msdx.i/x-18oct.b
Inst ID: msdx.i

(PPTV) RL

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL	
64 4-Ethyltoluene	130.86	131.82	128.94	126.98	135.28	138.12	137.57	132.79	4.30	13.50	100
65 1,3,5-Trimethylbenzene	60.57	59.85	58.70	59.08	62.12	60.42	60.97	60.25	1.16	3.64	20
66 Octane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
67 1,2,4-Trimethylbenzene	65.34	60.66	60.58	60.06	64.07	65.36	65.46	63.08	2.52	7.93	20
68 1,3-Dichlorobenzene	61.11	65.94	57.11	57.88	61.24	59.29	59.89	60.35	2.90	9.12	20
69 1,4-Dichlorobenzene	64.94	69.47	59.19	56.73	62.20	61.54	61.28	62.48	3.70	11.64	20
70 Benzyl Chloride	53.84	53.34	60.60	60.20	59.87	63.26	60.82	61.70	1.70	5.33	100
71 1,2-Dichlorobenzene	65.29	63.83	60.93	59.55	62.11	64.33	61.53	62.51	2.05	6.43	20
72 1,3-Dichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
73 Dibromomethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
74 1,2,4-Trichlorobenzene	87.81	94.36	87.07	88.33	93.21	93.42	86.52	90.11	3.40	10.67	50
75 Hexachlorobutadiene	75.02	75.01	74.77	72.75	78.10	72.56	72.81	74.43	1.97	6.19	50
76 Naphthalene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

Report Date : 10-Apr-2002 10:59

Air Toxics Ltd.

INITIAL CALIBRATION DATA

Start Cal Date : 04-APR-2002 11:34
End Cal Date : 05-APR-2002 12:46
Quant Method : ISTD
Origin : Disabled
Target Version : 3.50
Integrator : HP RTE
Method file : /chem/msdx.i/x-04apr.b/to14044f.m
Cal Date : 10-Apr-2002 10:07 lford
Curve Type : Average

Full Sim Curve
04-10-02 LF

NK 4/12/02

Calibration File Names:

Level 1:	/chem/msdx.i/x-04apr.b/x040405.d	2nd Source	X040509.d	12.5ml	5.0
Level 2:	/chem/msdx.i/x-04apr.b/x040407.d	RLV	X040403a.d	25.0ml	.05
Level 3:	/chem/msdx.i/x-04apr.b/x040408.d	RLV	X040404a.d	5.0ml	.1
Level 4:	/chem/msdx.i/x-05apr.b/x040505.d	RLV	X040405a.d	10.0ml	.2
Level 5:	/chem/msdx.i/x-05apr.b/x040506.d	RLV	X040406a.d	1.0ml	0.1
Level 6:	/chem/msdx.i/x-05apr.b/x040507.d	RLV	X040407a.d	5.0ml	0.5

	Compound	0.02000 Level 1	0.10000 Level 2	1.000 Level 3	5.000 Level 4	10.000 Level 5	20.000 Level 6	RRF	% RSD	
1	Propylene	*****	*****	*****	*****	*****	*****	*****	*****	<-
2	Dichlorodifluoromethane/Fr12	4.99435	4.65723	4.95745	4.62931	6.22350	6.16344	5.27088	13.856	
3	Freon 114	3.70792	3.65708	3.87624	3.63810	4.70247	4.72974	4.06692	13.391	
4	Freon 22	*****	*****	*****	*****	*****	*****	*****	*****	<-
5	Chloromethane	2.52746	1.86719	2.23299	2.08721	2.77697	2.77748	2.37022	15.818	
6	Vinyl Chloride	1.03752	1.23545	1.60648	1.55597	2.07256	2.05741	1.59423	26.369	
7	1,3-Butadiene	*****	1.17827	1.33588	1.34694	1.78975	1.80292	1.49075	19.243	
8	Dimethyl Ether	*****	*****	*****	*****	*****	*****	*****	*****	<-
9	Bromomethane	1.57614	0.94546	1.18284	1.12658	1.18244	0.99713	1.17010	18.026	
10	Chloroethane	0.86309	0.73684	0.83302	0.84008	1.14168	0.84947	0.87736	15.623	
11	Trichlorofluoromethane/Fr11	4.93597	5.79888	4.96355	5.01277	6.17423	7.64439	5.75496	18.382	
12	Freon 113	3.73387	3.48078	3.70624	3.48838	4.53668	4.39311	3.88984	11.827	
13	Acrolein	*****	*****	*****	*****	*****	*****	*****	*****	<-
14	1,1-Dichloroethene	3.01576	2.74665	3.04390	2.92883	3.88265	3.81066	3.23807	14.924	
15	Acetone	*****	3.40871	3.15615	3.05479	4.12509	4.10235	3.56942	14.383	
16	Ethanol	*****	*****	*****	*****	*****	*****	*****	*****	<-
17	2-Propanol	*****	*****	*****	*****	*****	*****	*****	*****	<-
18	Carbon Disulfide	*****	3.77099	4.22472	4.04542	5.17994	5.25642	4.53550	16.180	
19	1-Pentene	*****	*****	*****	*****	*****	*****	*****	*****	<-
20	Pentane	*****	*****	*****	*****	*****	*****	*****	*****	<-
21	Methylene Chloride	*****	3.23260	2.31076	2.17963	2.84399	2.79238	2.67107	16.051	
22	MTBE	*****	3.68152	4.65389	4.68093	6.51679	6.48827	5.20488	24.042	
23	trans-1,2-Dichloroethene	*****	1.88089	2.10429	2.08324	2.74989	2.66293	2.31385	16.207	
24	Hexane	*****	2.46774	2.86668	2.93581	3.96231	3.89348	3.22524	20.659	
25	1,1-Dichloroethane	3.10903	2.73851	3.06058	2.94915	3.90321	3.78062	3.25688	14.504	

Pooled % RSD 17.0221

Report Date : 10-Apr-2002 10:59

Air Toxics Ltd.

INITIAL CALIBRATION DATA

Start Cal Date : 04-APR-2002 11:34
End Cal Date : 05-APR-2002 12:46
Quant Method : ISTD
Origin : Disabled
Target Version : 3.50
Integrator : HP RTE
Method file : /chem/msdx.i/x-04apr.b/to14044f.m
Cal Date : 10-Apr-2002 10:07 lford
Curve Type : Average

Compound	0.02000 Level 1	0.10000 Level 2	2.000 Level 3	5.000 Level 4	10.000 Level 5	20.000 Level 6	RRF	% RSD	
26 Vinyl Acetate	+++++	1.93793	2.17388	2.19051	2.94198	2.90250	2.42936	18.980	
27 2-Butanone	+++++	2.90261	3.69799	3.76086	5.27555	5.32007	4.19142	25.415	
28 cis-1,2-Dichloroethene	2.56203	2.31178	2.66428	2.55564	3.39982	3.28734	2.79682	15.751	
30 Tetrahydrofuran	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
31 Chloroform	3.69495	3.24468	3.56835	3.36505	4.49785	4.35462	3.78757	13.750	
32 Cyclohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
33 1,1,1-Trichloroethane	3.90143	3.62498	4.05314	3.83046	5.15974	5.08920	4.27649	15.702	
34 Carbon Tetrachloride	3.92305	3.97306	4.30624	4.13882	5.63400	5.57929	4.59208	17.367	
36 1-Hexene(1)	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
(2)	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	<-
37 Benzene	1.22000	1.08803	1.13457	1.06154	1.40514	1.33684	1.20769	11.524	
38 Heptane	+++++	0.37877	0.46209	0.48099	0.69433	0.70503	0.54424	27.022	
39 1,2-Dichloroethane	0.68442	0.57955	0.58719	0.52299	0.69236	0.67913	0.62360	11.403	
41 Trichloroethene	0.55010	0.50280	0.50503	0.47086	0.62064	0.59106	0.54008	10.668	
42 1,2-Dichloropropane	0.34524	0.29757	0.32157	0.30543	0.40533	0.38945	0.34410	12.981	
43 1,4-Dioxane	+++++	0.16532	0.20740	0.20659	0.29205	0.28736	0.23176	23.990	
44 Bromodichloromethane	+++++	0.66394	0.73431	0.69174	0.94654	0.90577	0.78886	16.303	
45 cis-1,3-Dichloropropene	0.72934	0.54174	0.63228	0.61613	0.83774	0.81057	0.69463	16.857	
46 4-Methyl-2-pentanone	+++++	0.49430	0.60923	0.64892	0.99755	1.04558	0.75912	32.512	
48 Toluene	0.81582	0.70555	0.78341	0.79172	1.11990	1.07464	0.88184	19.450	
49 trans-1,3-Dichloropropene	1.56675	1.07905	1.27382	1.19165	1.49802	1.47465	1.34747	14.429	
50 1,1,2-Trichloroethane	0.99747	0.90492	0.93018	0.83168	0.99606	0.91704	0.92956	6.697	
51 2-Hexanone	+++++	1.26080	1.20632	1.16618	1.67826	1.82446	1.42720	21.179	
52 Tetrachloroethene	1.29703	1.13115	1.15958	1.09412	1.33972	1.22878	1.20840	8.017	
53 Dibromochloromethane	+++++	1.22393	1.33677	1.23677	1.55189	1.47354	1.36458	10.610	
54 1,2-Dibromoethane	1.01592	0.97540	0.98936	0.91710	1.12916	1.07106	1.01633	7.360	
56 Ethyl Benzene	0.53564	0.44776	0.51937	0.51112	0.64633	0.63064	0.54847	13.656	
57 Chlorobenzene	1.45875	1.23939	1.29141	1.23107	1.60997	1.53649	1.39452	11.656	
58 m,p-Xylene	0.48679	0.40618	0.51381	0.50431	0.61157	0.56276	0.51424	13.572	
59 o-Xylene	0.37988	0.30836	0.40129	0.41050	0.50796	0.46846	0.41274	16.884	
60 Styrene	1.03383	0.54939	0.74429	0.77995	1.02712	0.97750	0.85201	22.749	
61 Bromoform	+++++	1.02246	1.20977	1.18412	1.55176	1.49184	1.29199	17.240	
63 1,1,2,2-Tetrachloroethane	1.26990	1.02739	1.15396	1.10280	1.46347	1.45147	1.24483	14.674	

Appendix C

BFB Tune Criteria & Check Mix

4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

AIR TOXICS LTD.

Method : TO-15-S (Sh)-SIM-Roy F. Weston (Evergreen Manor)

Compound	Rpt. Limit (ppbv)
Freon 113	0.020
Methylene Chloride	0.10
cis-1,2-Dichloroethene	0.020
1,1,1-Trichloroethane	0.020
Benzene	0.050
Trichloroethene	0.020
Toluene	0.020
Tetrachloroethene	0.020
Ethyl Benzene	0.020
m,p-Xylene	0.040
o-Xylene	0.020
Acetone	0.50
2-Butanone (Methyl Ethyl Ketone)	0.10
Chloroform	0.020

Surrogate	Method Limits
1,2-Dichloroethane-d4	70-130
Toluene-d8	70-130
4-Bromofluorobenzene	70-130

Control Copy #:___

@ Air Toxics Limited
STANDARD OPERATING PROCEDURE

RECEIVING, LOGIN, AND TRACKING OF SAMPLES

SOP #50

Revision Date:	03/09/01
Revision #:	3
Reason for Revision:	To meet NELAP requirements and add login section.

Updated By:				
	Signature	Print	Title	Date
Technical Review:				
	Signature	Print	Title	Date
QA Review:				
	Signature	Print	Title	Date
Laboratory Director:				
	Linda L. Freeman			Date

1.0 SCOPE AND APPLICATON

The purpose of this Standard Operating Procedure is to outline the procedures for receiving air samples, login of samples in the database and in the proper storage area, and tracking these samples through the laboratory.

2.0 RECEIPT OF SAMPLES

2.1 The sample receiving team assumes the custodial responsibilities associated with receiving, inspecting, and logging samples into ATL's ATLAS Sample Tracking, an internal sample tracking software program.

2.2 The policy establishes specific guidelines for sample acceptance, which are generally accepted practices under EPA, AFCEE, Army Corps of Engineers, Navy, and NELAC protocols.

2.3 When samples do not meet the established guidelines, discrepancies are documented and the client is notified. Discrepancies are also noted in the Narrative portion of the sample report.

2.4 The Sample Acceptance Policy is posted in the sample receiving area and copy is provided to field staff with every shipment of containers or media.

2.4.1 The following terms are used in defining the receipt condition of the samples:

- Good: No obvious damage, proper temperature/pressure
- Questionable: Evidence of possible shipping damage, custody seals not intact, received with improper temperature/pressure, COC form not received or incomplete
- Poor: Definite shipping damage, integrity of samples obviously compromised, broken sample media.

2.5 Sample Acceptance Policy

2.5.1 Samples received by Air Toxics Ltd. are relinquished following standard EPA approved guidelines. These include full and complete Chain-of-Custody documentation indicating: unique sample name, location, date and time of collection, collector's name, preservation type (if applicable), matrix, and any special remarks.

2.5.2 The chain-of-custody form is filled out in ink and indicates proper preservation and use of sample container specified by the method.

2.5.3 Each sample is labeled with unique, durable, and indelible identification and must be of adequate volume for the tests requested.

2.5.4 Never affix a label directly on a Summa™ canister. A tag is attached to each canister for this purpose.

2.5.5 Proper, full, and complete inspection and documentation is performed upon laboratory receipt in the following areas:

- evidence of container's physical damage
- status of the container's custody seal
- presence or absence of a chain-of-custody form
- incomplete or incorrect chain-of-custody form
- number of samples
- name of each sample
- sample collection date/time
- sample location
- name of the collector
- preservation type (if applicable)
- sample type (canister, XAD, DNPH etc.)
- sample tag information complete
- temperature (when applicable)
- pressure (canisters)
- presence of unlabelled samples
- presence of mislabeled samples
- presence of unused media
- method required trip blanks, field blanks, equipment blanks, field duplicates, or field spikes

2.5.6 Any sample discrepancies against the above criteria are documented on the Sample Discrepancy Form (Appendix A) and communicated to the client via Login Fax within 1 day of sample receipt.

2.5.7 The client is contacted by the project manager for discrepancies of a more serious nature, e.g.

- Custody seal on the outside of the container was broken.
- Chain-of-Custody Record was not received with sample(s).
- Analysis method(s) is(are) not specified.
- Sample(s) received out of holding time.
- Flow controller used - canister samples received at <2.5"Hg.
- Sample container (Tube/VOA vial) was received broken.
- Container for VOA analysis received with headspace.

- Tedlar Bag received leaking.
- Tedlar Bag received flat.
- Canister received with a leaky valve.
- Tedlar bag/canister received emitting a strong odor (sample cannot be analyzed).
- Initial laboratory Vacuum/Pressure does not match final field pressure

2.5.8 Documentation of client notification is included on the sample discrepancy form along with any instructions from the client on how to proceed.

2.5.9 Project managers complete this section and return the form to the receiving group to complete the login process. The form is archived in the Work Order (WO) folder.

2.5.10 Whenever there is any uncertainty of how the laboratory is to proceed or when the desired method is unclear, the receiving staff places the Login process ON HOLD and delivers the WO file to a Project Manager for follow-up.

2.5.11 The project manager contacts the client to clarify the situation. Phone calls between the project manager and the client are documented in the Client Services database.

2.5.12 The phone contact and client instructions to resolve the issue are logged into the database and a hardcopy report is placed in the WO folder.

2.5.13 The project profile is checked for client's financial status. If the financial status is "blacklisted" the project manager will be contacted and the samples placed on hold. A status of "Credit App missing" the client will be notified about the missing credit application on the login fax/e-mail cover page and the workorder folder is stamped "financial hold". The samples will proceed normally into the laboratory.

2.5.14 The folder is then returned to the Receiving team to complete the Login process. Air bills, packing lists, chain-of-custody records, and any other documentation that may accompany the samples are placed in the WO folder.

2.5.15 Laboratory malfunctions that may occur during or after sample receipt are documented via the laboratory Corrective Action system.

2.5.16 Examples of receiving problems which would necessitate a Corrective Action Request include:

- Sample was broken during handling.
- Hold time expired due to laboratory error.
- Canister sample pressurized with wrong type of gas.
- Sample placed "on hold" was released in error.
- Sample logged in for incorrect analysis method.
- DANGER tag was not affixed to an odiferous canister sample before sending to the lab.
- Canister was released and cleaned before second analysis method was run. Receiving did not affix the multiple analysis tag.
- Canister valve was left open following pressurization. Sample vented to ambient.

2.6 If a sample is received that may be hazardous to handle, the following procedures are used:

2.6.1 SUMMA™ Canisters and Tedlar Bags:

- Canisters or Tedlar bags which emit a strong odor, are immediately placed in a fume hood by the sample custodian.
- The Hazardous Materials Coordinator or member of the Safety Committee is contacted to assess the level of hazard associated with the sample(s) and to ensure proper handling.
- Any leaking Tedlar bag sample, which contains high levels of Hydrogen Sulfide, will not be analyzed. The client is notified immediately so that re-sampling may be scheduled if necessary.

2.6.2 Liquid and Sorbent Media Samples:

- The sample recipient always wears protective gloves while unpacking these types of samples.
- If a liquid sample is received in a leaking or broken container the Hazardous Materials Coordinator is notified for proper cleanup and handling of the sample.
- The Hazardous Materials Coordinator is also notified if a sample on sorbent media is received broken, to ensure proper handling.

2.6.3 After above inspections, receiving personnel fill out the COC form(s) accurately. After signing the COC in the "Received for lab" location (or any "Received by" location if it is not indicated) the Sample Recipient must follow the signature with "ATL". The correct time and date as well as the following information, given the circumstance, is filled in:

- If it is ATL's COC, the bottom portion of the COC, designated for the person responsible for opening the sample package (includes shipping and custody seal information), is filled in.
- If it is not ATL's COC, the "Custody Seals intact" and the "temperature" is stamped in ink on the bottom portion of the COC is used and the appropriate information is filled out.

Any discrepancies found by the sample receiving team member must be documented on a Sample Receipt Discrepancy Form (see Appendix A).

2.7 After the samples have been properly logged into ATLAS (the samples noted on the COC), the number of samples, media and analysis type, and significant dates (promised and expiration) are listed in the ATLAS database.

2.7.1 The following information describes both the sample holding times and how to set up the promised due date in the ATLAS/Sample receiving database: *(It is important to note that both due dates are referenced to working days unless otherwise specified.)*

HOLDING TIMES

Sulfur -->1 day (Tedlar bag) & 3 days (Silco can) from date of collection.
Tedlar Bags --> 3 days from date of collection.
Formaldehyde extraction --> 7 calendar days from the date of collection.
TO-8 extraction--> 48 hours from time of collection
TO-13 extraction, liquids, PUF's--> 7 days from the date of collection.
Canisters, Tubes, and Natural Gas bombs --> 14 days from date of collection
Charcoal Tubes→ 14 days from date of collection
Siloxane→ 14 days from date of collection.

- If the analysis is to be performed with a quick turn around time, the sample receiving personnel put all documents (COC, air-bill, bid/ship, etc.) into a red folder.
- If the analysis is to be performed on a standard turn around time, the documents are placed in a yellow folder.
- The date and turnaround time is written on the outside of the red folders so that one is able to distinguish rush work orders quickly.
- The receiving duty is prioritized based on the following questions when considering which samples to give a work order number first (in order of highest to lowest priority):

1. Are the samples previously expired or expiring the same day of receipt?
2. Do the samples have a short hold time?
3. Do the samples need to stay cold?
4. Is the analysis requested a rush turn?
5. If the samples can be run the day of receipt, does the lab need them quickly?
6. Is the analysis a standard turnaround? Do the samples have a long hold time?

3.0 THE LOGIN/FAX EMAIL

3.1 The login fax is sent only if the folder and information entered into ATLAS has been checked by a second receiving personnel and found to be correct. This is documented on the receiving/login checklist. (See Appendix B)

3.1.1 When Login is completed, a fax or e-mail is sent to the client to confirm receipt of samples. The Login fax has four parts:

Page 1: Cover page with discrepancies noted
Page 2: Log-in summary (sample names etc.)
Page 3: Reporting template showing referenced method, target compound list, and reporting limits
Page 4: Client's COC

3.1.2 A copy of either the login fax or e-mail is placed into the appropriate workorder folder.

3.1.3 Discrepancies are noted on the cover page using a template of pre-approved statements. QA is responsible for maintaining the approved template. Receiving staff electronically copy relevant statements from the template and onto the FAX cover page.

3.1.4 Document in ATLAS the date the login e-mail was sent.

4.0 *CREATING THE WORK ORDER*

4.1 When sample receiving receives the media, the project profile and shipping form is matched by scanning the canister bar codes or manually by matching company and project information.

4.2 The samples, in which the media did not originate at ATL are matched with a project profile by the client service's representative who has been in contact with the client.

4.3 The Sample Receiver must include the following items in each WO when it is created:

- Chain-of-Custody (COC)
- Air-bill
- Shipping Form*, Contacts*, Quotes*
- Project Profile
- Sample Receipt Summary
- Discrepancy Report (Pink Sheet)
- Receiving Report (ATL media only)
- Other documents (i.e., special receiving form)*
- *if applicable

4.4 It is critical for the sample receiver to search out information on any unknown situation when receiving samples using ATLAS (i.e. Contacts, Shipping, Sample tracking, Quotes) Faxes, E-mail, or any other significant source.

4.5 The sample receiver attaches a print out of the Project Profile to the inside front cover of the WO folder. The Project Profiles contains the specific information laboratory personnel will need in order to process the samples. This information includes general client information as well as any specific project requirements that differ from ATL's SOPs.

4.6 A unique number is assigned to a WO using the Sample Tracking log. For example, the first batch of samples received in January 2001 would be identified as **0101001**. The first **01** refers to the year, the second **01** designates the month, and **001** determines the batch number. The Sample Receiver then enters the WO number and other pertinent information when creating the workorder in ATLAS, making sure that all information is correct regarding the samples just received.

4.6.1 The following are guidelines to assigning WO numbers when multiple analyses are requested and/or multiple COCs are received for samples:

a. When multiple analyses are requested on given samples on the same COC(s), the work order is divided by adding a letter to the end of the WO number. For example, if three analyses are requested from samples to be assigned work order **9901001**, each given WO would be defined as **9901001A, 9901001B, and 9901001C**.

b. If multiple COCs are received for samples analyzed for the same analysis, client, and project, all COCs may be placed in one WO.

c. When multiple COCs are received and the samples on each COC are from different projects, there must be as many WOs assigned as the number of COCs.

4.7 Fraction numbers and specific QA/QC samples (laboratory duplicates, etc.) are assigned to the samples in a WO based on the information in the Project Profiles, and the following instructions:

- When samples of different media type are received on the same COC and each media has a different analysis – the work order is split and fraction numbers are assigned by sequentially numbering the samples in order starting from fraction (01A) for that specific media type.
- If samples are received of the same media type, on the same COC, and for multiple analysis only on some of the samples - fractions are assigned by numbering all the samples sequentially, and writing only the fractions that apply to each WO on the COC.
- VOST tubes are given the fraction "A" for the Tenax (front) tube and "B" for the Tenax/charcoal (back) tube. If there is an Anasorb tube present, the Anasorb is always combined with a Tenax tube to be analyzed properly. Sorbent tubes and charcoal tubes are labeled "A" as front tube and "B" as a back tube as well.
- For work orders involving an extraction, only TO-4 and TO-13 samples are assigned fractions. For other cases, samples are labeled the work order number only.
- Duplicate analysis is not possible for samples in VOST tubes and Carbotraps.
- Duplicate analysis is possible for VOST Condensates, but only if it is specified in the Project Profile.
- Composite samples or samples that are to be combined into one are given fractions starting with the first sample labeled fraction "A", and subsequent samples labeled the next letter. For example, if three Tedlar bags were composited, the fractions would be 01A, 01B, and 01C.

4.7 Any equipment returned on a Bid/ship must be checked in using the Canister Asset Tracking System (CATS). Any special circumstance regarding labeling and analysis of samples is always documented in the project profile.

5.0 SAMPLE STORAGE, TRACKING AND DISPOSAL

5.1 Samples that do not require extraction must be stored in the sample cage in the main laboratory. The Sample Receiver must assure that all samples are placed in the sample cage by the end of the day. Samples that do not require extraction but must remain chilled are placed in the appropriate refrigerator located at the back of the sample cage.

5.2 Those samples, which require an extraction, are placed in the formaldehyde refrigerator, extractions refrigerator, or VOST refrigerator (depending on analysis), see Table 1.

Table 1

Location in Sample Cage	Contents
Section A1	Tedlar Bags
Sections A2-A3	1L summa canisters
Sections B1-D4	6L Summa canisters, tanks, and vacutainers

Location	Freezers	Contents
Cage	4	Charcoal tubes for NIOSH

Location	Refrigerators	Contents
Extractions Lab	1	Semivolatile samples and preparation media
Cage	4	VOST samples only
	5	Water for headspace analysis
Extractions Lab	6	Semi-volatile extracts, PCB and PEST extracts, and any other samples which do not belong in the other refrigerators
	7	Aldehyde and Ketone samples and preparation media
	8	Extracted samples for B1F0011
Team Leader Office	DZ	TSP Filters/PM10 Filters

5.3 The Sample Receiver records samples into either the Internal Sample Tracking logbook or the extraction's lab logbook titled "Extractable Sample Tracking". Samples that have already been extracted when they are received are entered into the "Extract Tracking" logbook. When entering the samples, the Sample Receiver has to include the WO number and specific fractions, location of samples, name and date.

5.4 As the samples are removed for analysis, the responsible analyst/scientist documents each step in the Internal Sample Tracking logbook. The following procedures are used:

- When removing a set of samples the analyst records his/her initials and the time and date of removal from the secure area.
- As each sample is analyzed, it is logged into the instrument logbook indicating date of analysis and unique computer file number.

- Samples removed from the secure area but not currently being analyzed are still considered to be in the possession of the analyst/scientist.
- Following analysis, the analyst/scientist returns the samples to the secure area and documents this action in the appropriate logbook.
- Samples remain in the secure area until time of disposal. Disposal is indicated in the Internal Sample Tracking logbook.

List of Appendices

Appendix A ***Sample Receipt Discrepancy Report***
Appendix B ***Receiving Login Checklist***

Appendix A

Sample Receipt Discrepancy Report

Sample Discrepancy Report

Initiated By: _____ Date: _____ Given To: _____

Sections I - III must be filled out by the person initiating this sample discrepancy report.

I. Work order affected: _____

Sample(s) affected: _____

II. Discrepancies:

To be noted in Log-in FAX

- ❑ Chain of Custody Record (COC) improperly relinquished.
- ❑ COC was not filled out in ink.
- ❑ Number of samples on the COC does not match the number of samples that were received.
- ❑ Sampling date / time is not documented.
- ❑ Sample tags do not match the COC.
- ❑ Samples received at wrong temperature.
- ❑ Canister sample received at >15"Hg.
- ❑ Sample container (Tube/VOA vial) was received broken, however, sample was intact.
- ❑ No brass cap.
- ❑ Other

To be noted by Client Services Call (if deemed necessary) or by Laboratory Personnel

- ❑ Custody seal on the outside of the container was broken.
- ❑ Chain of Custody Record (COC) was not received with sample(s).
- ❑ Analysis method(s) is not specified on the COC.
- ❑ Sample(s) received out of holding time.
- ❑ Flow controller used - canister samples received at <2.5"Hg.
- ❑ Sample container (Tube/VOA vial) was received broken.
- ❑ Container for VOA analysis received with headspace.
- ❑ Tedlar Bag received leaking, or found to be leaking at the time of analysis.
- ❑ Tedlar Bag received flat, or found to be flat at the time of analysis.
- ❑ Canister received with a leaky valve, or found to be leaking at the time of analysis.
- ❑ Tedlar bag / canister received emitting a strong odor (sample cannot be analyzed).

❑ Initial laboratory Vac./Press. does not match final field pressure.
❑ VOST tube saturated, bag dilution is necessary.
❑ Sample loss due to instrument malfunction

❑ Other

III. Describe the discrepancy.

_____

_____

_____

_____

_____

_____

IV. Client Notification:

Person notified: _____ Verbally by: _____ Date: _____

Person notified: _____ Login fax by: _____ Date: _____

Client Instructions:

_____

_____

_____

_____

_____

_____

_____

_____

_____

Client Services representative to check one of the following:

At Log-in, include this information on the: ❑ LOG-IN FAX
❑ Report Narrative

Appendix B

Sample Receiving/Login Checklist

RECEIVING / LOGIN CHECKLIST

- ☐ Does the amount charged match the Project Profile?
  - ☐ Check the surcharge.
  - ☐ Check the Misc. charges.
- ☐ Is the correct Project and Project Number used?
- ☐ Report to and Bill to correct?
- ☐ Is the TAT correct? (Check the COC) and Project Profile.
- ☐ Does the narrative reflect sample received?
- ☐ Check the list:
  - ☐ Does it match the COC?
  - ☐ Does it match the Project Profile?
- ☐ Clients financial status.
- ☐ Are there any client specific requests on the COC? Have they been addressed?
- ☐ Is a CVP package required?
- ☐ Have the Duplicates been assigned?
- ☐ Is a Diskette required?
- ☐ 24 Hour Clock.
- ☐ Check Notes to Receiving.
- ☐ Pink Form?
  - ☐ Need attention by Client Services?
  - ☐ Addressed by login fax?
- ☐ Reviewed by:_____ Date:_____
- ☐ Please make changes, then OK.

Changes made by:_____ Date:_____

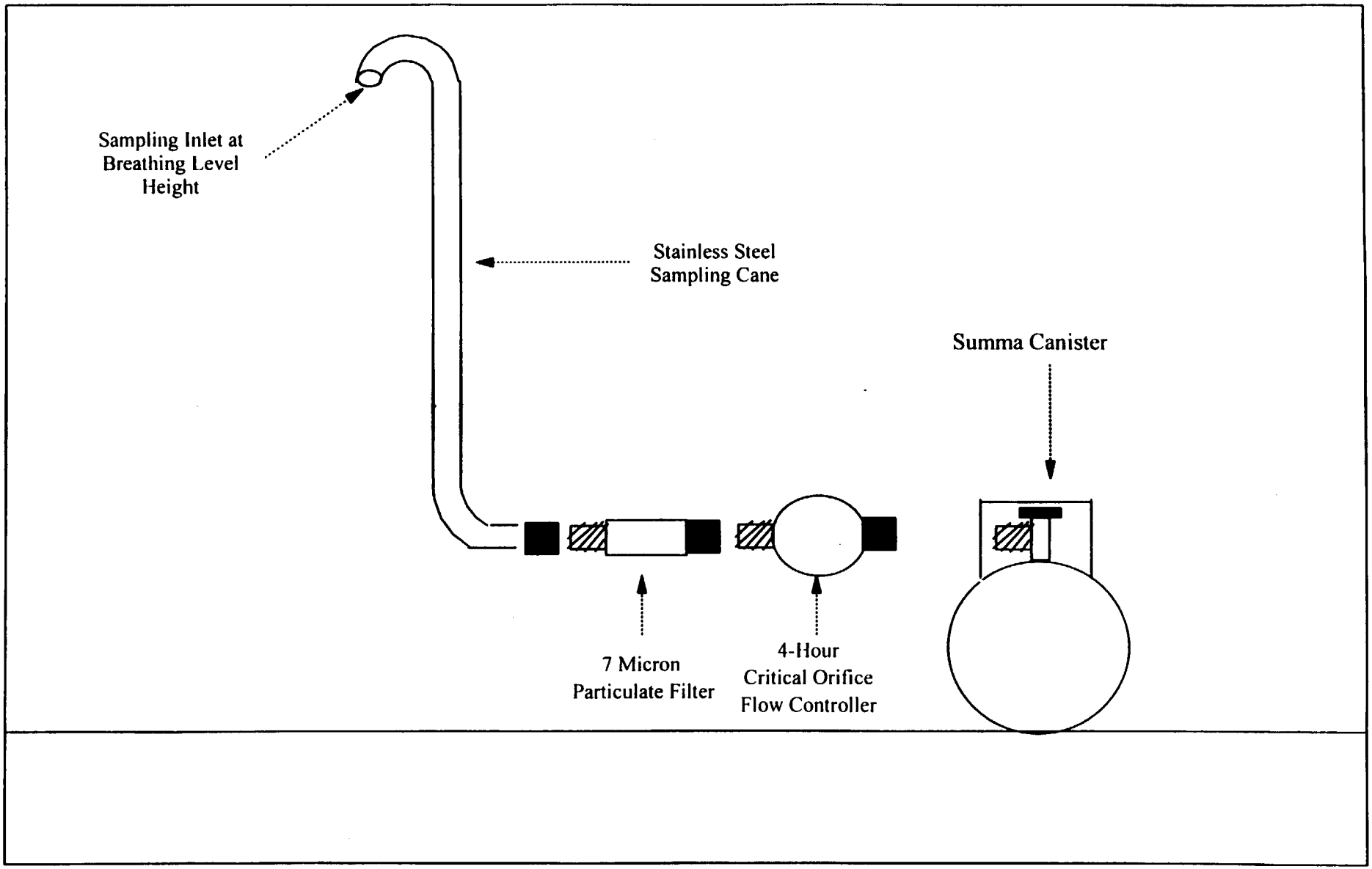
Other Explain: _____

Note: Complete each box with a "X" for completed or "NA" for Not Applicable. No box is to be left empty!

APPENDIX H

SAMPLING EQUIPMENT

Residential and Ambient Air Sampling Equipment



SUMMA Canister Sample Data Sheet

SAMPLE LOCATION	SAMPLE TYPE[1]	SAMPLE NUMBER	CANISTER NUMBER	SAMPLING PERIOD				VACUUM		COMMENTS/ OBSERVATIONS
				Start Date	Start Time	Stop Date	Stop Time	INITIAL ("Hg)	FINAL ("Hg)	
1= Residential, Soil gas, ambient, duplicate										

ADDITIONAL COMMENTS:

SAMPLES COLLECTED BY _____

DATE _____

DATA CHECKED BY _____

DATE _____